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RECYCLING OF WASTE OILS

NATIONAL OIL RECOVERY CORPORATION

PREPARED FOR
ENVIRONMENTAL PROTECTION AGENCY

JUNE 1975

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The objective of the work reported is the development of technology to recycle waste oils to useful products, without producing undesirable wastes. Both crankcase and other waste oils were studied in the laboratory and in a 1000 barrel per day vacuum distillation process operated by National Oil Recovery Corporation in Bayonne, New Jersey. Plant operations demonstrated that vacuum distillation is a suitable process for producing fuels from a wide variety of waste oils. Laboratory and engineering studies showed that the distillate side product produced from crankcase waste oil could be catalytically hydrotreated to produce a lube with good odor, color, and stability characteristics. Overall, the vacuum distillation/hydrogen treatment process for re-refining waste oils holds great promise. The distillation bottoms, containing high concentrations of lead and other metals, can be used as a fuel in secondary lead smelting. Pretreatment and chemical reduction agents show promise in refining, but additional laboratory and evaluation work is required.

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RECYCLING OF WASTE OILS

By

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FOREWORD

Man and his environment must be protected from the adverse effects of pesticides, radiation, noise and other forms of pollution, and the unwise management of solid waste. Efforts to protect the environment require a focus that recognizes the interplay between the components of our physical environment -- air, water, and land. The National Environmental Research Centers provide this multidisciplinary focus through programs engaged in

- studies on the effects of environmental contaminants on man and the biosphere, and

- a search for ways to prevent contamination and to recycle valuable resources.

This report covers attempts to develop an economical way of rerefining waste oils that might otherwise be wasted to usable fuel products, attempts to minimize pollution, and incidentally permits recovery of lead values.

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Director
National Environmental
Research Center, Cincinnati

ABSTRACT

The objective of the work reported is the development of technology to recycle waste oils to useful products, without producing undesirable wastes. Both crankcase and other waste oils were studied in the laboratory and in a 1000 barrel per day vacuum distillation process operated by National Oil Recovery Corporation in Bayonne, New Jersey.

Plant operations demonstrated that vacuum distillation is a suitable process for producing fuels from a wide variety of waste oils. Laboratory and engineering studies showed that the distillate side product produced from crankcase waste oil could be catalytically hydrotreated to produce a lube with good odor, color, and stability characteristics. Overall, the vacuum distillation/hydrogen treatment process for re-refining waste oils holds great promise. The distillation bottoms, containing high concentrations of lead and other metals, can be used as a fuel in secondary lead smelting.

Pretreatment and chemical reduction agents show promise in re-refining, but additional laboratory and evaluation work is required.

This report was submitted in fulfillment of Contract Number 68-01-0177, by the National Oil Recovery Corporation, Bayonne, New Jersey, under the sponsorship of the Environmental Protection Agency.

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SECTION I

CONCLUSIONS

1. Extensive plant operations by NORCO have shown that vacuum distillation is a suitable process for producing low water content fuel from a wide variety of waste oils.

2. NORCO's plant vacuum distillation experience and development work on catalytic hydrogen treating have provided the basis for a profitable process to produce lube stocks from crankcase waste oils.

3. Lead-containing vacuum distillation bottoms from crankcase waste oil processing can be considered as a fuel and a source of lead in the secondary lead smelting industry, but additional full scale tests are required.

4. Lead in vacuum distillation bottoms from crankcase waste oil processing can be concentrated by additional processing to 30% lead, a material potentially useful for lead recovery.

5. Commercially available anti-fouling agents are useful for reducing vacuum distillation fouling problems.

6. Pretreatment of crankcase waste oil with two parts of butanol to one part of crankcase waste oil to increase yields and decrease fouling is a potentially attractive method for improving the vacuum distillation/hydrogen treating process, but development work is required.

7. Pretreatment of crankcase waste oil with low concentrations of amines, or possibly ammonia, may decrease vacuum distillation fouling, but development work is required.

8. Low concentrations of chemical hydrogenation agents, e.g., sodium borohydride, can reduce fouling problems, and could possibly replace catalytic hydrogen treating, but development work is required.

9. The use of raw distillate from vacuum distillation of crankcase waste oil as a diesel fuel without further refining is questionable at best.

10. An economical wastewater system for a vacuum distillation process requires that indirect condensers and mechanical vacuum pumps be used to minimize the quantity of contaminated water.

SECTION II

RECOMMENDATIONS

1. A large scale demonstration of a vacuum distillation/hydrotreating process to re-refine crankcase waste oil to high quality lube oil without producing waste products.

2. Comprehensive full-scale tests for using lead-containing vacuum distillation bottoms in secondary lead smelting.

3. Continued research and development on chemical reduction methods for re-refining crankcase waste oils.

4. Study and demonstration of chemical emulsion breaking systems to concentrate oil from high water content oil/water wastes, e.g., ballast and bilge wastes from oil tankers, oil tank cleaning wastes, oil spill recovery wastes, etc.

5. Federal support should be considered for these recommended programs.

SECTION III

INTRODUCTION

Recent estimates show that only 80 million gallons per year of re-refined lubricating oil is available and sold, as compared to over one billion gallons per year of waste lubricating oils generated in the U. S. Most of the remainder is used as a fuel, used for road oiling and dust control, or disposed of to the environment. This situation is both wasteful, because of the high cost of producing virgin lubes from special crude oils; and environmentally damaging, because of fine metallic particles (including lead from crankcase waste oils) emitted when burning many waste oils. Indiscriminate waste oil disposal is harmful to surface water and can also interfere with the operation of wastewater treatment plants.

One of the major reasons for this unfortunate lack of recycling is the absence of adequate technology which can produce high grade lubricating oils, while minimizing or eliminating wastes. Acid/clay treating, the most commonly used re-refining process, produces acid sludge and spent clay wastes. The acid sludge, a concentrated sulfuric acid/polymerized hydrocarbon/metal contaminated mixture is a highly undesirable waste, generally disposed of on land. Such disposal can lead to water pollution problems. The spent clay, which is much less hazardous, is also disposed of on land. The technology used for handling other waste oils, not suitable for lubricating purposes, is also generally inadequate.

The work discussed in this report was aimed at improving the technology of waste oil recycling so as to produce useful products while eliminating or minimizing wastes. It is an extension of an earlier program which showed that vacuum distillation is a promising method for processing waste oils. Both laboratory and commercial scale tests (1000 barrels per day and higher) have been conducted to meet the stated objective.

SECTION IV

PLANT OPERATIONS

The National Oil Recovery Corporation (NORCO) facilities in Bayonne, New Jersey accept a wide variety of waste oils for reclamation and recycle. These range from automotive crankcase waste oils to tank bottoms recovered during tank cleaning operations, oil recovered from wastewater treating systems, and oil recovered after spills. Most of the lubricating type oils, such as crankcase waste oil, is processed by vacuum distillation to produce cuts suitable for further processing into lube oils or suitable for fuel use. Most of the other waste oils received are contaminated principally by water. These are dried by vacuum distillation and blended into fuels.

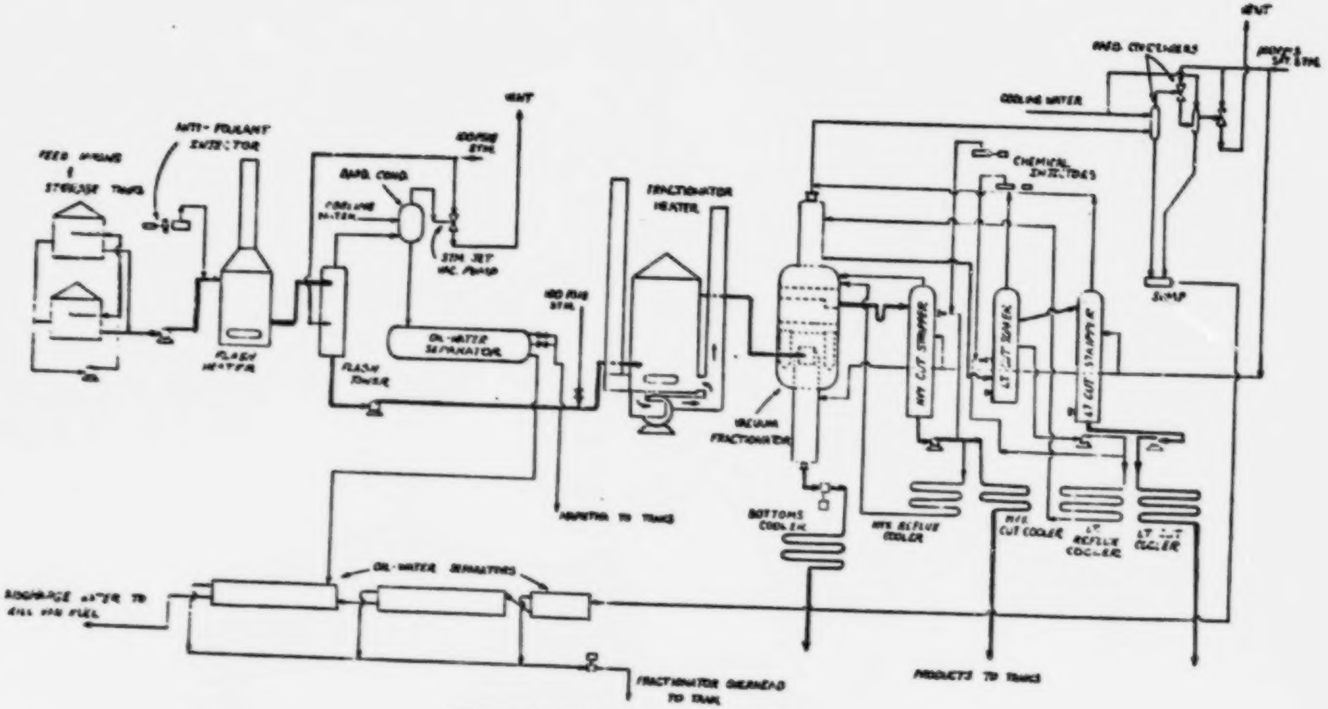
GENERAL PLANT DESCRIPTION

The NORCO plant equipment and operations were described in an earlier report.[1] Although some modifications have been made, the basic equipment and flows are similar to the descriptions in that report. An up-to-date flow sheet and a drawing of the vacuum distillation column are presented as Figures 1 and 2.

In addition to the basic equipment shown, NORCO maintains about two million gallons of feed and product storage. Detail on this storage is provided in Table 1. Waste oils are received primarily from private collectors, in trucks ranging in size from about 1400 gallons to 7000 gallons. Some waste oils are purchased in larger quantities and received in trucks arranged by contract.

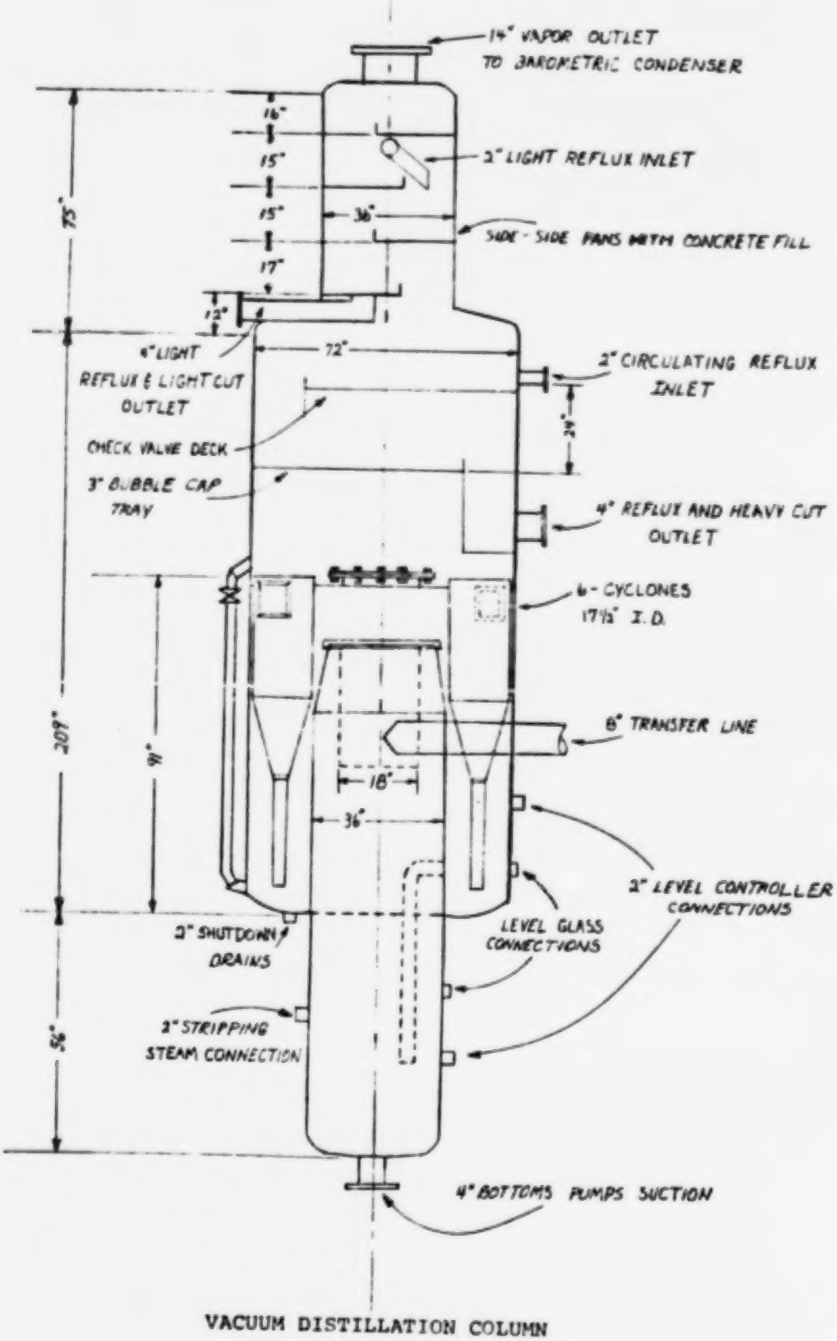
The waste oils are usually pumped directly into a large holding tank. Samples are taken for BS&W (bottom sediment and water) determination as necessary, depending on the oil source and the supplier.

A log of feed and products produced is given in Table 2. Operating data are provided later in this section for crankcase waste oil runs made in May 1973 and January 1974, and for thirty other waste oil runs made from November 1972 through May 1973.


FEED PUMPS
&
STORAGE TANKS
ANTI-FOULANT INJECTOR
FLASH HEATER
FLASH TOWER
BARO. COND.
COOLING WATER
STM JET VAC. PUMP
STM
VENT
OIL-WATER SEPARATOR
100 PSIG STM
NAPHTHA TO TANKS
FRACTIONATOR HEATER
VACUUM FRACTIONATOR
BOTTOMS COOLER
HVY CUT STRIPPER
HVY REFLUX COOLER
CHEMICAL INJECTORS
LT CUT TOWER
MED CUT COOLER
LT CUT STRIPPER
LT REFLUX COOLER
LT CUT COOLER
BARO. CONDENSERS
COOLING WATER
SUMP
VENT
STM
PRODUCTS TO TANKS
OIL-WATER SEPARATORS
DISCHARGE WATER TO
FOR FUEL
FRACTIONATOR OVERHEAD
TO TANK

CRANKCASE WASTE OIL PROCESSING FLOW SHEET

FIGURE 1


14" VAPOR OUTLET TO BAROMETRIC CONDENSER
2" LIGHT REFLUX INLET
SIDE - SIDE PANS WITH CONCRETE FILL
16"
15"
15"
17"
12"
36"
75"
4" LIGHT REFLUX & LIGHT CUT OUTLET
72"
2" CIRCULATING REFLUX INLET
24"
CHECK VALVE DECK
3" BUBBLE CAP TRAY
4" REFLUX AND HEAVY CUT OUTLET
6 - CYCLONES 17½" I.D.
8" TRANSFER LINE
209"
91"
18"
36"
2" LEVEL CONTROLLER CONNECTIONS
LEVEL GLASS CONNECTIONS
2" SHUTDOWN DRAINS
2" STRIPPING STEAM CONNECTION
56"
4" BOTTOMS PUMPS SUCTION

VACUUM DISTILLATION COLUMN

FIGURE 2

Table 1. PRINCIPAL NORCO TANKAGE
(as of April 1, 1974)

TANK NOS.	DIAMETER FT.	HEIGHT FT	NOMINAL CAPACITY OF EACH GALLONS	NORMAL SERVICE
1	40	30	280,000	Feed
2	40	30	280,000	Feed
3	40	30	280,000	Feed or Bottoms
100-109 (ten tanks)	15	42	55,000	Product
110-117 (8 tanks)	10	24	14,000	Product
129-130 (2 tanks)	20	20	47,000	Feed
205-209 (5 tanks)	15	42	55,000	Feed or Product
	16	16	24,000	Feed or Product
		Total	1,895,000 gallons*	

---

* Excludes miscellaneous small tanks and blending tanks.

Table 2. LOG OF FEED AND PRODUCTS

2/15-3/15/74 - 150,750 gal. waste fuel oil processed and 103,870 gal. saleable reclaimed fuel oil produced

1/15-2/15/74 - 55,840 gal. waste fuel oil processed and 38,640 gal. saleable reclaimed fuel oil produced

- 169,130 gal. crankcase waste oil processed and 161,680 gal. of saleable products produced including 45,000 gal. of 16.4°API bottoms for NL Industries test

12/15/73-1/15/74 - No runs

11/15-12/15/73 - 236,200 gal. waste fuel oil processed and 161,200 gal. saleable reclaimed fuel oil produced

10/15-11/15/73 - 295,200 gal. waste fuel oil processed and 212,200 gal. saleable reclaimed fuel oil produced

9/15-10/15/73 - 161,040 gal. waste fuel oil processed and 99,960 gal. of saleable reclaimed fuel oil produced

8/15-9/15/73 - 329,386 gal. waste fuel oil processed and 216,552 gal. saleable reclaimed fuel oil produced

7/15-8/15/73 - 379,048 gal. blended waste fuel oil (including 40°API recycled naphtha) processed and 280,839 gal. saleable reclaimed fuel oil produced

6/15-7/15/73 - 485,400 gal. waste fuel oil processed and 386,389 gal. saleable reclaimed fuel oil produced

5/15-6/15/73 - 58,750 gal. waste fuel oil processed and 44,850 gal. saleable reclaimed fuel oil produced

- 60,848 gal. crankcase oil processed to produce 15,029 gal. bottoms, 37,300 gal. dark lube oil, and 5,476 gal. fuel oil

4/15-5/15/73 - 288,396 gal. waste fuel processed and 226,964 gal. saleable reclaimed fuel oil produced

3/15-4/15/73 - 267,658 gal. waste fuel oil processed and 200,726 gal. saleable recalimed fuel oil produced

Table 2. (Continued) LOG OF FEED AND PRODUCTS

2/15-3/15/73	231,877 gal. waste fuel oil processed and 190,914 gal. saleable reclaimed fuel oil produced
1/15-2/15/73	346,085 gal. waste fuel oil processed and 280,498 gal. saleable reclaimed fuel oil produced
4/1/72-1/15/73	2,330,000 gal. waste oil received. Produced 1,572,000 gal. recycled fuel oil and 393,000 gal. of recycled crankcase oil products
11/7-12/7/72	349,441 gal. waste fuel oil processed and 316,532 gal. saleable reclaimed fuel oil produced
10/7-11/7/72	106,500 gal. waste fuel oil and 147,500 gal. waste lube oils processed
9/7-10/7/72	122,148 gal. waste fuel oil and 83,000 gal. crankcase waste oil processed
8/7-9/7/72	150,000 gal. waste fuel oil processed. About 50,000 gal. free water evaporated from the 250,000 gal. sludge, water, etc. received from Pottstown (Berks)
7/7-8/7/72	100,000 gal. fuel oil produced
6/7-7/7/72	100,000 gal. fuel oil produced from low sulfur oil (from Butterworthing) and some shore storage tank bottoms. 90,000 gal. fuel oil produced from oil from above sources plus about 30% barge tank bottoms containing about 40% water (pour point of 130°F reduced by the blending operation).
5/7-6/7/72	Both fuel oil and crankcase oil runs were made
4/7-5/7/72	Both fuel oil and crankcase oil runs were made
3/7-4/7/72	Several test runs were conducted

CRANKCASE WASTE OILS

When sufficient crankcase waste oil, at least 40,000 to 80,000 gallons, has been received and stored separately from other waste oils, a run is conducted. Five products are recovered during such a run:

Flash Tower Naphtha (#1)
Vacuum Distillation Naphtha (#2)
Light Distillate (#3)
Heavy Distillate (#4)
Vacuum Distillation Bottoms

Referring to Figure 1, the crankcase waste oil is pumped from a heated storage tank to a vertical coil heater, where it is heated to about 200-210°F. The heater coil is composed of 13-5ft. turns of 3 in. schedule 40 pipe. The oil leaving the furnace enters a 3 ft. diameter, 20 ft. high flash column which is designed primarily to dehydrate the raw oil. The flash column is fitted with a cyclone at the outlet to minimize particle entrainment, and a ½ in. screen at the bottom to prevent coarse solids from entering the bottoms pump.

The flash column vacuum (19-22 in. Hg) is generated by a single stage steam jet pump after a barometric condenser. The condensed water and the flash tower naphtha (NORCO #1) distilled overhead go to an oil/water separation tank from which the bulk of the naphtha is recovered. The contaminated water is discharged to the wastewater system which will be described later in this Section.

The bottoms, at about 160 to 200°F, is pumped to the vacuum fractionator furnace where the oil is heated and vaporized, reaching a temperature of 600-700°F. The furnace, fired by a single burner using plant fuel (usually naphtha), is rated at about 8 million BTU/hr. fired, with a duty of about 4-5 million BTU/hr. The furnace contains 84-12 ft., 4 in. O.D. tubes with a 1/8 in. wall thickness.

The oil leaving the furnace enters the vacuum fractionator, where vacuum distillation naphtha (#2), light distillate (#3), heavy distillate (#4), and vacuum distillation bottoms cuts are recovered. Both light distillate and heavy distillate reflux streams are returned to the vacuum fractionator at 150-200°F; the light distillate to the top of the column, and the heavy distillate to the upper distillation tray.

The fractionator (Figure 1) is maintained at about 27 in. Hg vacuum by a two stage steam jet with barometric condensers. The oil/water mixtures from the condensers are drained to a common sump and then to oil/water separators for recovery of vacuum distillation naphtha and water purification.

The light and heavy distillates withdrawn from the vacuum distillation column each pass through stripping columns. The liquid is removed from the bottom of each column and split into reflux and product streams. The reflux and distillate product streams, as well as the bottoms product, are separately cooled in pipe coils submerged in a tank through which cooling water from the Kill Van Kull is circulated once-thru. The light distillate product cooler contains 12-19 ft. 3 in. sections of 2 inch welded schedule 40 pipe with return bends; each of the other four coolers contains 6 sections. Each of the products, whose temperatures may vary from about 130°F for the light distillate to about 280°F for the bottoms, is then pumped to an appropriate storage tank. The reflux streams are returned to the fractionator as previously noted.

May 1973 Run

A crankcase oil run was made primarily to produce an experimental quantity of 11.0 - 12.0° API bottoms for testing by NL Industries to determine the practicality of using the bottoms for fuel and as a lead source.

This crankcase oil run was interrupted three times by mechanical difficulties: to renew the horizontal run of the 4 in. flash tower barometric condenser cooling water drain pipe; to renew the drive belt on the fractionator heater burner; and finally to renew the drive shaft of the burner. Difficulty with the flow of light cut reflux was experienced during the last part of the run because of tarry accumulation and displacement of concrete filler on the top liquid-washed side-to-side pan at the top of the fractionator. None of the above problems was necessarily the result of running crankcase oil, since all of this equipment is used also for other waste oils. However, tar accumulation is expected from crankcase oil distillation.

An anti-foulant from Nalco Chemical Co. (D-59C08)was injected into the crankcase oil charge (ahead of flash tower furnace) and into the light and heavy dark lube oil cuts at the rate of 50 ppm by weight at each point. The results were very similar to those obtained when an anti-foulant from Exxon was injected in 1969.[1] Fouling in heater tubes appeared to be reduced. Also fouling in the light and heavy lube oil cooling coils was somewhat reduced. The color of these two products was slightly darkened, to about L7.5 to 8.0 (ASTM) from a normal L7.0. The tarry material settling in the bottoms of bottles of samples of the lube distillates remains fluid and does not solidify and tenaciously adhere to the bottom of bottles, as occurs without anti-foulant injection. The odor seemed to be somewhat reduced. The Nalco anti-foulant is considerably less viscous than that from Exxon. The lubricators satisfactorily injecting the Exxon anti-foulant are not dependable injecting the less viscous Nalco anti-foulant.

The yields and operating conditions for this run are reported in Tables 3 and 4. The bottoms produced had a gravity of 11.9°API and were found by NL Industries to be satisfactory for a full scale trial as a fuel in a reverberatory furnace.

Table 3. MAY 1973 CRANKCASE WASTE OIL RUN FLOW RATES

Start: May 17, 1973 Complete: May 25, 1973
(interrupted by mechanical problems)
Onstream Time: 39.2 hours

	Gravity °API	Total Gallons	Flow GPH	Yield Vol. %
FEED	23.4	60,864	1554*	--
Flash Tower Naphtha (#1)	46.1	913	23.3	1.5
Vac. Dist. Naphtha (#2)	33.2	5,478	139.9	9.0
Light Distillate(#3)	31.1			
Heavy Distillate(#4)	30.3	37,310	952.6	61.3
Bottoms	11.9	15,027	383.7	24.7
Water + Loss+	--	2,136	54.5	3.5
		60,864	1,554.0	100.0

UTILITIES

Cooling Water	200 GPM
Steam for Stripping	0
Steam to vacuum pumps	700 lbs/hr
Steam to fractionator	0
Steam to bottoms pump	50 lbs/hr
Steam to tank heaters	155 lbs/hr
Total steam produced	905

* 888 barrels/day
+ unaccounted for

Table 4. MAY 1973 CRANKCASE WASTE OIL RUN AVERAGE CONDITIONS

	Temperatures, °F
Flash heater inlet	72
Flash heater outlet	200
Flash tower vapor outlet	161
Flash tower bottom	160
Fractionator heater inlet	158
Fractionator heater outlet	670
Fractionator flash zone	630
Fractionator bottom	610
Fractionator top	250

	Pressures
Flash tower	20-21 in. Hg. vacuum
Fractionator bottom	27.2 in. Hg. vacuum
Flash heater inlet	12 psig.
Fractionator heater inlet	54 psig.
Steam boiler	105 psig.

January 1974 Run

A brief but successful crankcase waste oil run was conducted in January 1974 during which special attention was given to obtaining process data useful for design purposes. The run started on January 22 and was shut down four days later on January 26 because of a shortage of feed. Before the run started, the following vessels were opened, cleaned, inspected and closed: fractionator; flash tower; light and heavy product reflux accumulators; and light and heavy product stripper.

Feed and product inspections are summarized in Table 5 and 6. They show high chlorine values in all fractions, and surprisingly high lead contents in the light fractions. This lead may be due to volatile lead compounds, or to fine particulate entrainment. A previous analysis of a blend of the two NORCO vacuum sidestream distillates (#3 and #4) showed a lead content of 2 ppm.

Average run conditions are shown in Tables 6-10. Yields are estimated in Table 11. As can be seen, this particular run provided a high yield of vacuum distillation bottoms at the expense of lube stock yield, providing a light, relatively low viscosity, low lead bottoms easier to handle for the NL Industries combustion tests.

Two anti-fouling additives were used during this run. Nalco Additive No. D-59C08 was injected into the cold crankcase oil charge prior to the flash heater, and sodium borohydride was injected into the light and heavy distillate product reflux accumulators. Additive rates were as follows:

Nalco D-59C08	50 ppm by weight
Sodium Borohydride	275 ppm by weight (total $NaBH_4$) as a 12 weight % solution in 40% NaOH solution

In order to detect the effect of the sodium borohydride, the vacuum distillation tower was opened and inspected after the run. The following observations were made (see Figure 2):

Tower Bottom

In normal runs the deposit is hard. During this run the deposit was softer and looked like asphalt.

Cyclone Deck

There was very little deposit in this section. The sodium borohydride appears to have inhibited deposit formation.

Top of 6 ft. Section

There were fewer deposits than previously noted.

Top of Light Cut Section

A hard rubber like deposit came out of this section from the top tray. This deposit is considered unusual.

Light End Stripper

The bottoms from the accumulator showed a dark, heavy residue. In previous runs the residue had been hard, but in this run it was soft and greasy.

None of the deposits appeared to be serious, but the run was too short to draw definite conclusions.

However, simple sodium borohydride injection in this run did not provide the color improvement found during research studies reported in Section V.

Table 5. INSPECTIONS FOR CRANKCASE OIL RUN - JANUARY 1974

SAMPLE DESCRIPTION	Feed	Flash (#1) Naphtha	Vac.Dist.(#2) Naphtha	Vacuum Distillation Light Distillate (#3)						Vacuum Distillation Heavy Distillate (#4)					Vac. Dist Bottoms
	1/25-8AM	1/25-12PM	1/25-8AM	1/25-4PM	1/25-12noon	1/25-8AM	1/24-4AM	1/23-6PM	1/22-8PM	1/26-4PM	1/25-4PM	1/24-4AM	1/23-6PM	1/22-8PM	1/25-8AM
API Gravity @ 60°F	24.0	36.9	35.1	31.00	31.44	31.33	30.96	30.75	31.55	29.75	30.00	29.90	29.73	29.93	16.8
Carbon, Wt. %	85.37	85.82	85.78	86.18	--	--	--	86.39	--	--	86.61	--	86.58	--	85.87
Hydrogen, Wt. %	12.83	12.27	12.35	13.52	--	--	--	13.3	--	--	13.22	--	13.31	--	12.38
Sulfur, Wt. %	0.33	--	0.21	0.19	--	--	--	0.19	--	--	0.16	--	0.17	--	0.61
Nitrogen, Wt. %	0.11	0.03	0.63	None	--	--	--	0.02	--	--	0.01	--	None	--	0.54
Chlorine, Wt. %	0.56	15.2	0.81	0.63	--	--	--	0.09	--	--	0.16	--	0.06	--	0.27
Bromine, Wt. %	--	--	--	--	--	--	--	--	--	--	--	--	--	--	0.21
Ash, Wt. %	1.57	--	--	0.11	0.097	0.030	0.034	0.062	0.153	0.15	0.084	0.014	0.099	0.124	4.40
Water, Wt. %	4.0	--	Trace	0.1	Trace	Trace	Trace	0.2	0.2	trace	trace	trace	trace	trace	trace
Lead, ppm	6800	185	35	16	-	--	--	9	--	--	14	--	10	--	16,000
Pentane Insol., Wt. %	7.25	0.02	0.02	0.28	0.33	0.013	0.041	0.019	0.81	0.20	0.13	0.081	0.23	0.20	6.03
Viscosity @ 100°F, sec.				78	80	76	110	104	100	259	240	266	296	250	160 (122°F)
ASTM Color	--	1.1	2.75	4.2	4.2	4.1	5.0	4.5	4.2	8+	8.0	7.0	6-8	8+	--
Flash, °F	--	--	112	245	240	240	255	250	245	410	420	425	430	420	440
Corrosion, 3 hrs @ 212°F	--	--	--	Passes/1B	--	--	--	Passes/1B	--	--	Passes/1B	--	Passes/1B	--	--
Con. Carbon, %	--	--	--	0.06	--	--	--	--	--	--	0.16	--	0.06	--	--
Neut. No., mg KOH/g	--	--	--	1.32	--	--	--	0.81	--	--	0.11	--	0.22	--	--
Pour Point (ASTM), °F	--	--	--	--	--	--	--	5	--	--	--	--	15	--	--
IBP, °F	--	--	--	416	--	--	--	--	--	--	292	--	--	--	--
10% Recovery	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Recovery, %	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Cracked, °F	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--

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Table 6. LABORATORY DISTILLATION DATA
(ASTM Distillation D-86)

	LIGHT DIST. (#3) 1/25/74-4 PM	HVY. DIST. (#4) 1/25/74-4 PM
Initial Boiling Point, °F	368	414
5% Recovery	535	665
10%	606	678
12%	--	680
20%	647	690
30%	--	692
40%	678	695
50%	688	698
60%	690	--
70%	694	--
80%	707	--
Final Boiling Point	712	714
% Recovery	86	55

Table 7. JANUARY 1974 CRANKCASE WASTE OIL RUN

AVERAGE TEMPERATURES

	°F
Flash heater inlet	51
Flash heater outlet	200
Flash tower vapor outlet	152
Flash tower bottom	152
Fractionator heater inlet	121
Fractionator heater outlet	607
Fractionator flash zone	593
Fractionator bottom	588
Fractionator top (vapor outlet)	203
Vapor space above hvy reflux inlet	406
Heavy cut at additive mixer	528
Light cut at additive mixer	238
Heavy cut at stripper inlet	542
Light cut at stripper inlet	243
Bottoms Cooler Inlet	547
Bottoms Cooler Outlet	283
Heavy Reflux Cooler Inlet	506
Heavy Reflux Cooler Outlet	176
Light Reflux Cooler Inlet	230
Light Reflux Cooler Outlet	156

Table 7 (Continued) JANUARY 1974 CRANKCASE WASTE OIL RUN

	°F
Light Distillate Cooler Inlet	234
Light Distillate Cooler Outlet	131
Heavy Cooler Outlet	206
Line to 1st Stage Vacuum Pump	42
Line to 2nd Stage Vacuum Pump	135
Cooling Water Intake	41
Cooling Water Discharge	54
Ambient	40

Table 8. JANUARY 1974 CRANKCASE WASTE OIL RUN

AVERAGE PRESSURES

Flash Heater Inlet	1.4 psig
Flash Heater Outlet	14.2 in. Hg vacuum
Fractionator Heater Inlet	32 psig
Fractionator Heater Outlet	20.8 in. Hg vacuum
Fractionator Flash Zone	28.1 in. Hg vacuum
Fractionator Bottom	28.1 in. Hg vacuum
Vacuum Fractionator Barometric Condenser Outlet	28.4 in. Hg vacuum
Light Cut Drawoff	28.9 in. Hg vacuum
Heavy Cut Drawoff	28.3 in. Hg vacuum
Heavy Stripper Inlet	28.1 in. Hg. vacuum
Light Stripper Outlet	28.2 in. Hg vacuum
Bottoms Cooler Inlet	6 psig
Bottoms Cooler Outlet	3 psig
Heavy Reflux Cooler Inlet	45 psig
Heavy Reflux Cooler Outlet	43 psig
Heavy Distillate Cooler Outlet	43 psig
Light Reflux Cooler Inlet	41 psig
Light Reflux Cooler Outlet	8 psig
Light Distillate Cooler Inlet	31 psig
Light Distillate Cooler Outlet	30 psig
Flash Barometric Condenser Outlet	23.4 in. Hg vacuum

Table 8. (Continued) JANUARY 1974 CRANKCASE WASTE OIL RUN

Steam Boiler	103 psig
Water Pump	25 psig

Table 9. JANUARY 1974 CRANKCASE WASTE OIL RUN
STEAM CONSUMPTION

	lbs./hr.
Flash Tower Vacuum Pump	131
Fractionator Vacuum Pumps	456
Bottoms Pump	69
Heavy Cut Stripper	0
Light Cut Stripper	0
Fractionator	0
Control Room	12
	668

Table 10. JANUARY 1974 CRANKCASE WASTE OIL RUN

MAJOR SOURCES OF POWER CONSUMPTION
220 Volts, 3 Phase

	Motor RPM	Amps	Hp.
Water pump	1755	64.8	25
Heavy cut pump	3450	26.8	10
Light cut pump(S)	1730	13.6	5
Light cut pump(N)	1760	27.0	10
Flash tower bottoms pump (Viking)	1140	15.0	5
Flash tower bottoms pump (Imo)	3500	8.4	3
Test separator pump (Moyno)	1170	22	7.5
Air compressor	1740	19	7.5
Feed pump	1155	22.4	7.5
Flash Heater Burner	3450	3.9	1.5
Fractionator Heater Burner	1750	8.8	3
Fractionator Heater Blower	1160	9.8	3
Compressor - South Boiler	3500	20	7.5
Burner - South Boiler	1730	2.0	½
Fuel Pump - North boiler	1160	5.4	1.5
Burner - North Boiler	3500	25.4	10
		294	108

Table 11. JANUARY 1974 CRANKCASE WASTE OIL RUN

Start: January 22, 1974 Stop: January 26, 1974
Onstream Time: 90 hours

	Total Gallons	Gallons/ hr.	Vol. %
Feed	169,000	1879*	--
Products			
Flash Tower Naphtha (#1)	1,000	11	0.6
Vac. Dist. Naphtha (#2)	36,800	409	21.8
Light Distillate (#3) Heavy Distillate (#4)	80,400+	893	47.5
Bottoms	45,000	500	26.6
Water (Flash Tower)	5,500	61	3.3
Loss (unaccounted for)	400	--	0.2
	169,100		100.0

* 1074 barrels per stream day.
+ Approximately 58% light distillate, 42% heavy distillate.

OTHER WASTE OILS

During the period covered by this Report, NORCO accepted waste oils from a multitude of sources. Considerable plant and laboratory effort was consumed in characterizing waste oils and in converting them to useful products. Difficulty in measuring water content of very wet oils was ocercome as shown in Table 12.

The following represents a few of the oil sources and characterizations. Additional data are provided in Appendix G, and in Section V.

- Oil stored in 20x20 South Tank (charge for run no. 9), 10-15% water, 8.5 to 29.5°API, depending on sampling point.
- Oil from tank cleaning, 33.8% H_2O, 0.9% sediment.
- Oil from an industrial separator, 1.4% H_2O, 0.74% sediment.
- Tank bottoms, 50.2% BS&W.
- Oil from a spill cleanup, 47.6% H_2O, 13.5% ash
- Oil from barge cleaning, 0-90% H_2O

The prinicpal methods used for upgrading the wide variety of waste oils received were settling in tanks, drying by distillation, and blending. Drying operations were conducted in the vacuum distillation column, bypassing the flash column used in crankcase waste oil operations. The vacuum column was generally run at about 22-26 in. Hg vacuum, and at a bottom temperature near that recommended on Table 13. The throughput was limited primarily by the water content of the oil being run.

Product characteristics were largely a function of the feed type. Extensive blending was used to produce saleable fuel oil. Table 14 provides an example of processed fuel in storage.

Data for 28 runs using blended waste oils are reported in Appendix K.

Table 12. WATER DETERMINATION METHOD FOR WASTE OILS

Determination of water content in many waste fuel oils cannot be satisfactorily obtained by the usual centrifuging tests, ASTM D1796 and ASTM D2709, because of formation of a gel which precipitates in the bottom of the centrifuge tube. Water in the oil apparently combines with various compounds in the oil to form the gel in a total volume which bears no direct relation to the volume of water. If the sample is diluted 1:1 with naphtha or kerosene and distilled in a manner somewhat like that described in the test ASTM D-95, the water content can be accurately and reliably determined. Modification of apparatus and procedure in distillation test ASTM D-95 provides rugged inexpensive apparatus and satisfactory procedure for determining water content over a very wide precentage range. The sample may be heated and distilled to the required temperature to drive over all water much faster than when following the procedure specified in distillation test ASTM D-86. Also the dilution with naphtha or kerosene may be varied according to viscosity and probably water content, judged from gravity and appearance.

Following are some typical determinations made in this way using a 50-50 mixture of waste oil and benzene.

Load No.	% H_2O	% Sediment	Total BS&W
1	66.0	3.0	69.0
2	76.6	3.0	79.6
3	73.3	3.0	76.3
4	50.0	3.0	53.0
6	70	3.0	73.0
7	90	1.3	91.3
8	66	3.0	69
27	60	2.0	62
48	60	1.0	61

Table 13. RECOMMENDED TEMPERATURES FOR DRYING WASTE FUEL OIL

Fractionator Vacuum in. Hg	Boiling Point of Water °F	Recommended Fractionator Bottom Temp., °F*
29	76.5	117
28	100	140
27	114	154
26	124.5	165
25	133	173
24	140	180
23	146	186
22	152	192
21	157	197
20	161	201
18	169	209
16	176	216
14	182	222
12	187	227
10	192	232
8	197	237
6	201	241
4	205	245
2	209	249
Atmospheric	212	252

*40°F over boiling point, °F (heater stack limited to 660°F and heater inlet limited to 62 psig.)

Table 14. TYPICAL FUEL PRODUCT IN TANKAGE DECEMBER 1972

Gravity	25.0°API
Flash Point P.M. C.C.	206°F
Viscosity, S.U. @ 100°F	350 secs.
Viscosity S.F. @ 122°F	24.9 secs.
Pour Point	minus 25°F
Sulfur, ASTM D1552	0.37%
Water & Sediment	0.3%
BTU/gallon	144,632
Ash	0.04%

OPERABILITY PROBLEMS AND SOLUTIONS

Operability problems encountered with crankcase waste oils in a vacuum distillation type process have been discussed in a previous report.[1] Mechanical and other problems encountered because of the wide variety of waste oils processed in recent operations are discussed in Appendix A, along with discussion of solutions to many of the problems considered.

The crankcase waste oil problems are related primarily to oil instability, the tendency of the oil to form polymeric materials, especially at elevated temperatures but also at moderate temperature. This is true for the distillation products as well as the raw oil. The chemistry of this problem and methods of pretreatment are considered in Section V.

As discussed earlier in this section and in the earlier report,[1] anti-fouling additives are hlepful even though not totally effective. These may be expected to cost less than 0.1¢ per gallon of oil for 50 ppm of the Nalco or similar types, but the cost of the borohydride used in the January 1974 crankcase oil run would exceed 1¢ per gallon. Insufficiant experience was accumulated to determine optimum concentration or justification for anti-fouling additives.

The importance of careful design to minimize serious fouling during crankcase waste oil operations cannot be overemphasized. For example:

1. Tower internals should be simple and should not allow accumulation of liquids. Mist eliminators tend to foul and have been replaced by cyclonic type mist separation in both the flash column and the vacuum fractionator. Spray decks in the upper section of the fractionator were filled with concrete to prevent liquid accumulation and tar formation.[1]

2. Furnaces must be designed to minimize extreme fouling caused by high tube metal temperature, even at the expense of higher capital cost. Flue gas recycle practiced by NORCO and internal furnace modification to prevent excessive radiation have improved operation.

3. Furnaces, heat exchangers, and product coolers must be designed for easy access to allow fast cleanout when fouling does occur, or spares must be provided.

4. Product accumulators are useful where settling of solids can occur without causing serious fouling problems such as in lines, valves, or pumps. A screen is used in the bottom of the flash column to avoid such problems.

These design approaches are useful in improving operability, but do not contribute to improved lube distillate quality. This can be accomplished only by treatment, such as with clay or with hydrogen.

Other waste oils may cause problems similar to crankcase oil, but other problems also arise as discussed in Appendix A. The most serious of these are solids deposition throughout the system, but especially in furnace tubes, and erosion due to sand and other foreign materials which contaminate many "garbage oils." Here, design solutions such as suggested above, are important as is filtration prior to processing and anti-erosion design. The elimination of sharp turns, the use of extra-hard materials, and especially the replacement of elbows which plugged tees have been found to be helpful. The foreign material tends to accumulate in the plugged branch of the tee, providing an erosion resistant surface.

POLLUTION CONTROL

Because most processing systems are closed and because the waste oils normally encountered are high boiling and low in sulfur, air pollution has not been a problem in NORCO's operations. Very little odor is noted from the oil/water separator area, from tank vents, or from the vacuum system. Therefore, it is not believed to be necessary to provide air pollution controls in the type of operation conducted to date. As will be discussed in Section VI, hydrogen treatment will produce small quantities of gaseous emission which may require some form of control.

On the other hand, wastewater from the processing plant must be monitored and controlled for oil and possibly other pollutants. The four primary sources of oil emissions are:

1. Oil vaporized when distilling or drying crankcase or other waste oils which contaminates steam condensate from steam jet vacuum pumps and steam stripping, cooling water used in barometric condensers, and water which is also vaporized from wet oils.
2. Oil remaining in water removed from tanks where primary oil/water separation takes place.
3. Oil which leaks from coolers into the cooling water.
4. Spills and leaks in process areas which contaminate surface runoff waters.

In the present NORCO operation, the greatest volume of oil contaminated water arises from the first source, as shown below:

	GPM
Flash tower barometric condenser (normally used only for crankcase waste oil)	70
Vacuum distillation barometric condensers	180
Product coolers (contaminated only when leaking)	50
Boiler blowdown	minor
Tank withdrawals and runoff water	variable

All wastewater is processed through the oil/water separation system.

Barometric condenser effluents undergo primary oil/water separation in tanks to recover the bulk of the oil present. The contaminated water effluent from these tanks, with other wastewaters, pass through two oil/water separators in series. These separators each have a holding capacity of about 7300 gallons, providing a total residence time of 49 minutes when the flow rate is 300 GPM.

Oil separation from discharged water appears to be highly variable as shown in Tables 15-17. Oil and phenol contamination appear to be higher when processing crankcase waste oil than when other waste oils are processed. At times, the oil content of the intake water from the Kill Van Kull appeared to be extraordinarily high. The wastewater discharge data reported here are consistent with previously reported information on other waste oil processing plants.[8]

A General Electric Model OPC-50 oil/water separator was tested during the January 1974 crankcase waste oil run as to suitability for oil removal from wastewaters. The separator is a gravity type designed to achieve total laminar flow of the wastewater from the input manifold, through baffles, and through a combination of specially configured coalescing plates and packs. With a flow head of approximately two feet to maintain capacity flow, the unit operates at atmospheric pressure with essentially no pressure drop from inlet to outlet. While the wastewater flows through the separator horizontally, the oil adheres to the plates and moves vertically through the specially configurated plate banks. When the oil reaches the liquid surface level in the separator, it is automatically skimmed off by a passive float device that can also control the oil layer thickness to insure that water-free oil is removed from the separator. The separator can be run under an inert gas blanket, and a coalescing media pack can be provided for final polishing, but these options were not used in this test. A sketch of the separator and a more detailed description may be found in Appendix H.

The GE OPC-50 separator provided a nominal residence time of 14 minutes at 100 GPM (neglecting internals). The test flow scheme is shown in Figure 3. Data obtained at 60 to 100 GPM flow rate through the GE separator are shown in Table 17. The separator removed 90-98% of the oil, but the high inlet loading prevented really effective cleanup. The GE separator could be considered for final cleanup after conventional gravity separators.

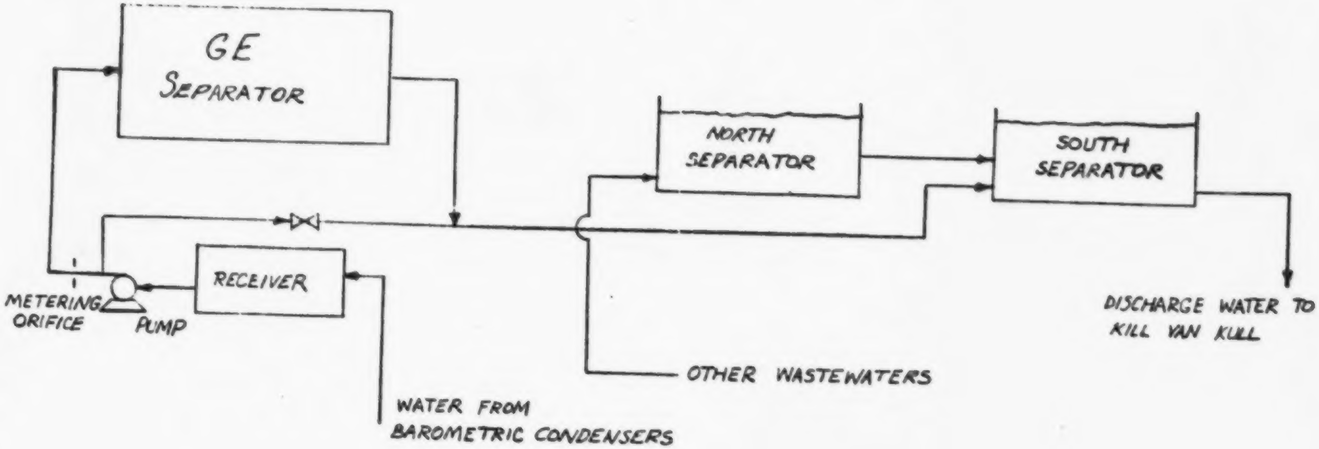
Table 15. WASTEWATER QUALITY WHEN PROCESSING MISCELLANEOUS WASTE OILS

Date (1973)	Sample	Oil ppm	Phenol ppm	pH	TOC ppm
2/14-15	CW In*	< 20ND	<0.05ND	6.8	
	Out+	209	5.1, 0.1	6.15	350,360
2/21-23	CW In	-	0.15	7.72	
	Out	-	0.8	6.65	17,425,380
2/26-27	CW In	-	-	7.2	
	Out	-	-	6.75	312,575
3/5-6	CW In	-	-	7.1	
	Out	-	-	6.6	
4/9-10	CW In	-	<0.05ND	7.25	
	Out	-	16	5.2	
4/25-26	CW In	< 1ND	<0.05ND	6.9	
	Out	2.4	5	6.15	
4/30-5/1	CW In	-	<0.05ND	7.15	
	Out	-	5	6.0	
5/31	CW In	-	<0.05ND	6.85	
	Out	-	2	5.95	
May Crank-case Oil Run	CW In	-	<0.05ND	7.15, 7.1	
	Out	-	14, 10	5.45, 5.3	

* From Kill Van Kull. CW = cooling water.
+ Leaving 2nd oil/water separator

Table 16. WASTEWATER OIL AND GREASE CONTENT DURING MISCELLANEOUS FUEL OIL RUNS - JUNE-JULY 1973

	Oil & Grease, ppm	
	In	Out
June 1973	103	124
	<1 ND	<1 ND
	13	90
	38	70
	68	31
	<1 ND	2
July 1973	6	16
	97	54
	6	10
	5	21
	8	35
	<1 ND	2
	9	14
	6	13


GE
SEPARATOR
NORTH
SEPARATOR
SOUTH
SEPARATOR
RECEIVER
METERING
ORIFICE
PUMP
WATER FROM
BAROMETRIC CONDENSERS
OTHER WASTEWATERS
DISCHARGE WATER TO
KILL VAN KULL

FLOW DIAGRAM FOR GE SEPARATOR TEST

FIGURE 3

Table 17. WASTEWATER ANALYSES - JANUARY 1974 CRANKCASE WASTE OIL RUN

	Sample Date	Sample Time (All PM)	pH	mg/l			
				Total Suspended Solids	Phenols	Oil Content	TOC
Cooling Water Inlet	1/23/74	8	6.8	8	0	0	18
GE Separator Inlet	"	"	6.4	11	9	2100	560
GE Separator Outlet	"	"	6.0	5	10	40	430
Discharge to Kill*	"	"	6.5	14	10	1200	350
Cooling Water Inlet	1/24/74	2	7.2	23	0	0	18
GE Separator Inlet	"	"	6.4	36	10	2500	420
GE Separator Outlet	"	"	6.5	53	14	450	380
Discharge to Kill*	"	"	6.5	47	6	1250	310
Cooling Water Inlet	1/25/74	2	7.5	6	0	0	17
GE Separator Inlet	"	"	6.7	26	12	2000	380
GE Separator Outlet	"	"	6.5	20	12	200	365
Discharge to Kill*	"	"	6.7	35	6	500	425
Cooling Water Inlet	1/26/74	2	7.1	47	0	0	11
GE Separator Inlet	"	"	6.6	81	12	2800	575
GE Separator Outlet	"	"	6.7	24	12	100	377
Discharge to Kill*	"	"	6.9	50	6	700	312

GE Separator Outlet Temperature approximately 44°F

* Kill Van Kull

SECTION V

RESEARCH STUDIES

The research conducted in this program was aimed primarily at improving methods of recycling crankcase waste oils. They consisted of:

- laboratory and field studies of methods for pretreatment, designed to avoid problems in processing crankcase waste oils by distillation;
- laboratory studies of bottoms, and other lead containing fractions;
- laboratory studies of catalytic hydrogen treatment to improve color, odor, and other properties of lube distillates;
- laboratory studies of chemical reductions with hydrides to improve color, odor, and other properties of lube distillates; and
- diesel engin tests on a distillate fraction.

In addition, considerable laboratory work was done to characterize both crankcase and other waste oils, and water effluents. These data are covered in Section IV.

PRETREATMENT

Pretreatment experiments were conducted both to try to separate existing sludge and metals in crankcase waste oil, and to eliminate precursors to further sludge formation during processing. These precursors appear to be products of crankcase oil reactions with blowby gases, such as nitrogen oxides. Precursors can be related to nitrogen oxide compounds present in the used oil.[2] The use of caustic and centrifugation pretreatment has been described previously.[8] In this work, centrifugation experiments with and without solvents succeeded in separating metals and sludge, and treatment with amines appeared to improve oil stability.

Centrifugation

As shown in Table 18, some separation of sludge and water does occur from crankcase waste oil by settling This can be enhanced by dilution with naphtha. However, the process is slow and incomplete. The difficulties of filtration, as an alternative, are described in Appendix C.

The centrifugation experiments in Tables 19-21 clearly show that, although high speed centrifugation succeeds in removing sludge and water, the recovered oil remains unstable and new sludge is formed at 250°F from precursors (Table 21). As expected, separated sludge contains a wide variety of metals as shown by semi-quantitative spectographic analysis. Quantitative analysis for Pb, Ba, Zn, and Ca showed 2.4, 1.4, 1.1, and 0.9 weight percent respectively (Table 22). Commercial centrifuge experiments described in Appendix D were relatively unsuccessful because a force of only about 6000 x G was obtainable and because of difficulty in solids discharge. However, the use of butanol, which will be described under Solvent Treatment, did improve solids separation to some extent.

Solvent Treatment

The following solvents were screened to test their ability to coagulate and precipitate impurities in crankcase waste oil: methanol, ethanol, n-propanol, isopropanol, n-butanol, isobutanol, pentanol, cyclohexanol, toluene, methyl ether, methylethylketone, acetone, amyl alcohol, glycerol, n-heptanol, hexanol, 2-furaldehyde, furfurol, dodecanol, tetraethylenepentamine, phenol, n-octanol, iso-octanol, 2-aminoethanol, hexane and naphtha (Tables 22 and 23). For the data in Table 24, solvent ratios of 1 to 4 volumes per volume of raw crankcase waste oil were shaken in a separatory funnel and allowed to settle for several days. The light solvents such as methanol and ethanol showed little precipitation power. The more promising solvents, as determined by visual observation of precipitation power, were studied further to determine the relative amounts of precipitate produced. In the 10,000 x G one hour experiments, the supernatant solvent phases appeared very bright for cyclohexanol, butanol, and octanol, as compared to a dark oil supernatant liquid where no solvent was used.

Table 18. SEPARATION OF SLUDGE AND WATER FROM CRANKCASE OIL BY SETTLING

	Vol. % Water and Sludge on Standing*				
	1 Hour	3 Hours	24 Hours	48 Hours	Wt. % Residue^{+}
Crankcase Oil	-	-	-	12	-
Crankcase Oil/Naphtha (1:1)	8.3	21.7	25.0	-	2.0
Crankcase Oil/Naphtha (2:1)	10.0	-	20.0	-	2.2

* Volume % based on oil only (specific gravity of water layer = 1.016)

+ After 72 hrs. standing, decanting water layer, 1 hr. centrifugation at 10,000 G. Based on oil only. Oil layer dark-bright.

Table 19. CENTRIFUGATION OF CRANKCASE OILS

Charge:*	CCO-1	CCO-1	CCO(A)	CCO(B)	CCO(C)	CCO(D)
Solvent	Naphtha	Naphtha	-	-	-	-
Solvent/CCO$^+$	1/1	1/1	0/1	0/1	0/1	0/1
Centrifugation, G	10,000	5,000	10,000	10,000	10,000	10,000
min	60	60	60	60	60	60
Wt. % water	-	-	15.0	1.5	1.0	1.0
Wt. % residue	2.00	2.02	1.5	1.3	1.4	1.6
Phases	-	-	-	-	-	-
Top Phase	-	-	-	-	-	-

Charge:*	CCO(E)	CCO-2	CCO-2	CCO-2	CCO-2	CCO-1
Solvent	-	-	Naphtha	Phenol	Phenol	Naphtha
Solvent/CCO$^+$	0/1	0/1	1/4	1/10	1/5	1/1
Centrifugation, G	10,000	10,000	10,000	10,000	10,000	10,000
min	60	60	60	60	60	30
Wt. % water	0.5	-	-	-	-	-
Wt. % residue	1.8	1.3	1.5	1.8	1.7	1.92
Phases	-	2	3	3	3	-
Top Phase	-	Dark	Dark	Bright	Bright	-

* CCO-1 = raw crankcase oil from NORCO tankage (batch 1)
CCO-2 = raw crankcase oil from NORCO tankage (batch 2)
CCO(A), CCO(B), etc. = raw crankcase oil from supplier A, B, etc.
+ wt. ratio
# Preheated to 130°F prior to centrifuging

Table 19. CENTRIFUGATION OF CRANKCASE OILS (Continued)

Charge: *	CCO-1	CCO-2	CCO-2	CCO-2	CCO-2	CCO-2
Solvent	Naphtha	n-Butanol	n-Butanol	-	Naphtha	Naphtha
Solvent/CCO$^+$	1/1	1/1	1/4	0/1	1/4	1/2
Centrifugation, G	5,000	5,000	5,000	5,000	5,000	5,000
min	30	30	30	60	60	60
Wt. % water	-	-	-	-	-	-
Wt. % residue	1.96	1.54	1.27	0.69	1.55	1.93
Phases	-	-	-	2	3	3
Top Phase	-	-	-	-	-	-

Charge:*	CCO-2$^\#$	CCO-2$^\#$	CCO-2$^\#$	CCO-2	CCO-2	CCO-2
Solvent	-	Naphtha	Naphtha	-	Naphtha	Naphtha
Solvent/CCO$^+$	0/1	1/4	1/2	0/1	1/4	1/2
Centrifugation, G	5,000	5,000	5,000	3,000	3,000	3,000
min	60	60	60	60	60	60
Wt. % water	-	-	-	-	-	-
Wt. % residue	0.91	1.43	1.70	0.69	1.10	1.37
Phases	-	-	-	2	3	3
Top Phase	-	-	-	Cloudy	Cloudy	Slightly Cloudy

Table 20. SEPARATION OF SLUDGE AND WATER FROM CRANKCASE OIL BY CENTRIFUGATION

Charge: Oil/Naphtha = 1:1

	3,000 G		5,000 G		10,000G	
	30 min.	60 min.	30 min.	60 min.	30 min.	60 min.
Volume % Water	0	0	30	30	30	30
Color of Oil	-	-	--- Dark Brown ---		--- Dark Bright ---	
Residue, wt. %*	1.9	2.0	2.2	1.8	2.3	1.9

* Based on oil only

Table 21. CONSECUTIVE HEATING AND CENTRIFUGATION OF CRANKCASE SUPERNATANT OILS

Original charge--Raw crankcase waste oil
Heating Phase--250°F for 60 minutes (with stirring)
Centrifugation--32,000 G for 60 minutes
Heating + Centrifugation + Residue Washing + Decanting
= 1 Cycle

Cycle No.	Wt % Residue[+]
1	1.3 (8 tests/1.26-1.40)
2	0.10 (4 tests/0.08-0.11)
3	0.05 (4 tests/0.048-0.055)
4	0.13 (4 tests/0.12-0.14)
5	0.09 (4 tests/0.09-0.10)
6	0.10 (4 tests/0.09-0.10)
7	0.05 (4 tests/0.04-0.06)
8	0.05 (4 tests/0.04-0.07)

[+] Based on supernatant liquid charged to cycle indicated.

Table 22. ANALYSIS OF SOLID SLUDGE FROM CENTRIFUGATION OF RAW CRANKCASE OIL

PPM by Semiquantitative Spectrographic Analysis
(ppm by quantitative analysis)

7000-70,000	700-7000	70-700	7-70	0.7-7	<0.7
Ca (8,900)	P	B	As	Bi	Be
Pb (23,620)	Mg	Mn	V	Ag	
	Fe	Sn	Zr		
	Si	Cr	In		
	Al	Ni	Co		
	Cu	Mo	Bi		
	Zn (10,600)	Ti			
	Ba (13,700)	Sr			

Not Detected: Sb, Nb, Cd, Na, K, Hg

Procedure: Approximately 40 g. samples of used crankcase oil were centrifuged in 50 ml. conical tubes to obtain a sludge that was oil wetted (Sorvall Model SS-1 high speed centrifuge). The oil wetted solids were further washed with pentane to arrive at dry solids which were analyzed (further drying at 100°C overnight yielded a 30% weight loss). Dry solids approximately 2% of original oil.

Table 23. SOLVENT MISCIBILITY TESTS
1 part solvent: 1 part raw crankcase oil
at room temperature

1. Hexane - completely miscible

2. Butanol - 2 phases--dark sediment on bottom, light red layer on top

3. Pentanol - 1 phase

4. Methyl ether - 2 phases--80% bottom dark, 20% top red

5. Toluene - 1 phase

6. Methyl ethyl ketone - 2 phases--dark deposit on sides, black oil on top

7. Methanol - 2 phases (very distinct)--top yellow layer

Table 24. RESIDUE PRODUCED BY SOLVENT TREATING OF RAW CRANKCASE WASTE OIL

Centrifugation at 32,000 G for 1 hr.

Solvent/Oil Ratio	wt. % Residue				
	0	1:1	2:1	3:1	4:1
No Solvent*	4.1	-	-	-	-
isopropanol*	-	4.1	4.4		5.4
isobutanol*	-	3.6	4.2	-	6.5
n-butanol*	-	4.4	4.1	-	17.7
2-aminoethanol#	-	15.3	16.4	18.5	21.0
cyclohexanol	-	8.4	7.5	6.8	-

Centrifugation at 10,000 G for 1 hr.

Solvent/Oil Ratio	wt. % Residue				
	0	1:1	2:1	3:1	4:1
No Solvent	3.3	-	-	-	-
isopropanol	-	-	-	-	6.3
isobutanol	-	-	-	-	8.3
n-butanol	-	3.7	4.4	6.9	7.0

Centrifugation at 10,000 G for 1 hr.+

Solvent/Oil Ratio	wt. % Residue				
	0	1:1	2:1	3:1	4:1
n-butanol**	-	2.3	2.2	2.6	2.6
cyclohexanol#	-	2.2	2.2	2.6	3.3
n-heptanol	-	2.1	2.1	-	-
hexanol	-	2.1	2.4	-	-
2-furaldehyde	-	1.7	1.9	-	-
furfurol	-	2.2	2.2	-	-

Centrifugation at 10,000 G for 30 min.

Solvent/Oil Ratio	wt. % Residue				
	0	1:1	2:1	3:1	4:1
No Solvent	2.6	-	-	-	-
dodecanol	-	2.7	2.7	-	-
octanol	-	2.9	3.3	2.6	-

---

* Residue pentane washed

+ Residue pentane washed twice followed each time by 10,000 G for 15 min.

# 3 phases present

** 4 phases present

The high residue observed with 2-aminoethanol (Tables 24 and 29) could indicate either a high precipitation efficiency or reaction of the solvent itself. Most likely, at least some reaction occurs.

The alcohol group shows real promise for precipitation. A process based on isopropanol has been proposed.[11] In the present work, n-butanol was pursued further, including the engineering feasibility study reported in Section VI, and some simple treating experiments shown in Tables 25-28. Treatment with butanol results in an oil with a clear reddish cast and a sweet odor. The odor can be eliminated by treatment with hot water or vacuum evaporation, but subsequent high temperature distillation restores a "burnt" odor.

Work with various amines, shown in Tables 29-32, indicates that these can accomplish precipitation similar to the solvents previously discussed, but with lower concentrations. Odor seems to be reduced by amine treatment, probably by reaction with carbonyls. Color is sometimes intensified, possibly by reduction of nitrogen oxide compounds forming azo groups. Inexpensive amines or ammonia could be considered as agents for sludge precipitation, but further investigative work is required.

LEAD RECOVERY FROM CRANKCASE WASTE OILS

Lead concentrates can be recovered from tank bottoms and pretreatment precipitates (Table 33), or from vacuum distillation bottoms. The distillation bottoms, or unvaporized portion of the crankcase waste oil fed to the vacuum distillation column, contains virtually all of the unprecipitated metals and high boiling polymerized fractions of the oil, as well as some hydrocarbons which could be considered potentially valuable as lubricating stock. The oil serves as a carrier for the impurities, but in actual fact should be minimized to the extent possible. To minimize the quantity of bottoms, it is necessary to operate at as high a vacuum and as high a temperature as possible, limited by cracking in the preheat furnace and in the distillation column.

Previous studies have shown that impurities in crankcase waste oil, such as nitrogen and oxygen, also tend to concentrate in the bottoms fraction.[3] These data are reproduced as Figures 5-8. Properties of typical bottoms fractions are provided in Section IV.

The crankcase waste oils available at the time this work was done (most gasoline leaded) contained on the order of 1% lead, with significant quantities of calcium, zinc, and barium. Based on taking a 5 to 20 percent bottoms cut from crankcase waste oil feed, the bottoms lead content would then be about 5 to 20 weight percent.

Table 25. BUTANOL TREATING EXPERIMENT

Charge:	70	ml. raw crankcase waste oil
	120	ml. butanol
	30	ml. pentane
	20	ml. acetone
	240	

Results: Immediate dropout of sludge. Centrifuged for 1 hr.

Distillation: Mixture heated in water bath with vacuum applied to flask. 50 ml. oil recovered from flask dark (6+ color) with only a very faint smell of alcohol. Alcohol recovered had a yellowish tinge.

Chromatographic Treatment: 10 ml. of above oil passed over 10 g. of a 24/40 granular material (Georgia-Tennessee Mining & Chemical Co., Harrison, N.J.). No change in color.

Table 26. BUTANOL TREATING EXPERIMENT

Charge:	40 ml. butanol
	160 ml. raw crankcase waste oil

Results: Solids separated in centrifuge

Distillation: Mixture heated in water bath to 65°C water temperature at 27 in. Hg vacuum. 68 ml. left in flask after a few hours. Alcohol remaining in flask as detected by odor.

Table 27. BUTANOL TREATING EXPERIMENT

Charge:	160 ml. butanol (80 Vol. %) 40 ml. raw crankcase waste oil (20 Vol. %)
Results:	About 2% by weight solids separated in centrifuge
Distillation:	About 130 ml. of solvent came off at 110-118°C. As temperature rose in the distillation an acrid smell was noted in the oil. An IR examination showed acid present which had been previously absent (butyric)

Table 28. DISTILLATION OF n-BUTANOL-CRANKCASE OIL MIXTURE

Charge: 250 ml of 1:1 n-butanol/crankcase oil mixture after centrifuging for 30 min. at 10,000 G

Liquid Still Temp, °C	Vapor Temp, °C	Cut, ml.
99	87	1st drop over
102	94	17.5 (5 ml. bottom layer)
105	95	12.5 (3 ml. bottom layer)
109	96	14.5 (2.5 ml. bottom layer)
115	103	17.0 (1.0 ml. bottom layer)
119	110	20.5 (no bottom layer)
120	113	24.6 (no bottom layer)
120	113	30.0 (no bottom layer)
120	113	30.5 (no bottom layer)
120.5	113	30.0 (no bottom layer)
125	115	20.5 (no bottom layer)
		217.6

(Some alcohol still in bottoms)
Added 250 ml. of fresh charge to bottoms

103	92	1st drop over	
107	94	25	Cumulative
109	98	50	
115	105	75	
118	110	100	
121	111	150	
123	111	175	
133	110	210	
		20 end	
		230 ml.	

Recovered 48 ml. of bottoms

Recovery = $\frac{217.6 + 230 + 48}{500} = 99\%$

Table 29. SOLVENT TREATMENT WITH 2-AMINO ETHANOL

	Residue After Centrifugation At 10,000 G for 30 min.			
Solvent/Oil Ratio	1:1	0.5:1	0.1	0
Number of Phases	3	3	3	2
Vol. % - Top Phase	62.5	60	90	-
Vol. % - Middle	37.5	38	10	-
Wt. % - Residue	18.1	19.8	9.0	2.6

Table 30. TREATMENT OF SUPERNATANT LIQUIDS FROM CENTRIFUGATION WITH TETRAETHYLENEPENTAMINE

a. 1 part by wt. of naphtha to 2 parts crankcase oil centrifuged at 32,000 G for 30 min.
(Top layer = 1.58 ml/g. crankcase oil)

b. Top layer heated to 120°F with 1 part tetraethylenepentamine to 10 parts original crankcase oil charge and centrifuged at 32,000 G for 30 min.
(Top layer = 0.96 ml/g. original crankcase oil charge)

c. Residue washed with pentane twice
(% Residue, based on original crankcase oil charge, = 1.0)

Table 31. SOLVENT TREATMENT WITH 2-AMINO-ETHANOL

Oil Charge*	Distillate	Distillate	Distillate	CCO	CCO
Wt. Ratio, Solvent/ Oil Charge	1/10	1/4	1/2	1/10	0
Centrifuge Speed, G	5000	5000	5000	5000	5000
Centrifuge Time, min.	60	60	60	60	60
Separation					
Top, ml/g oil	1.01	1.04	0.99	0.65	0.70
Bottom, ml/g oil	0.04	0.15	0.25	-	0.27
Residue Treatment+	---Washed with 20 ml P/twice with 20 ml M/ twice with 20 ml 3:1 P:M/twice with P @ 5000 G, 15 min. --------				
Wt. % Residue	2.13	2.15	2.00	1.95	1.19

* CCO = raw crankcase waste oil
+ P = pentane; M = methanol; i-P = isopropanol

Table 31. SOLVENT TREATMENT WITH 2-AMINO-ETHANOL (Continued)

Oil Charge*	CCO	CCO	4:1 CCO/Naphtha
Wt. Ratio, Solvent Oil Charge	1/10	1/10	1/4
Centrifuge Speed, G	3000	5000	5000
Centrifuge Time, min.	30	30	60
Separation			
Top, ml/g oil	0.70	0.75	1.40, 0.90
Bottom, ml/g oil	-	-	-
Residue Treatment+	3 washes with 1:1 i-P:P/once with P @ 3000 G	washed with 1:1 i-P:P	washed 4 times with 1:1 i-P:P/once with P
Wt. % Residue	1.55	1.98	0.8, 2.0

Table 32. TREATMENT OF CRANKCASE OIL WITH DIETHYLENETRIAMINE (DET)

Charge: Raw crankcase oil

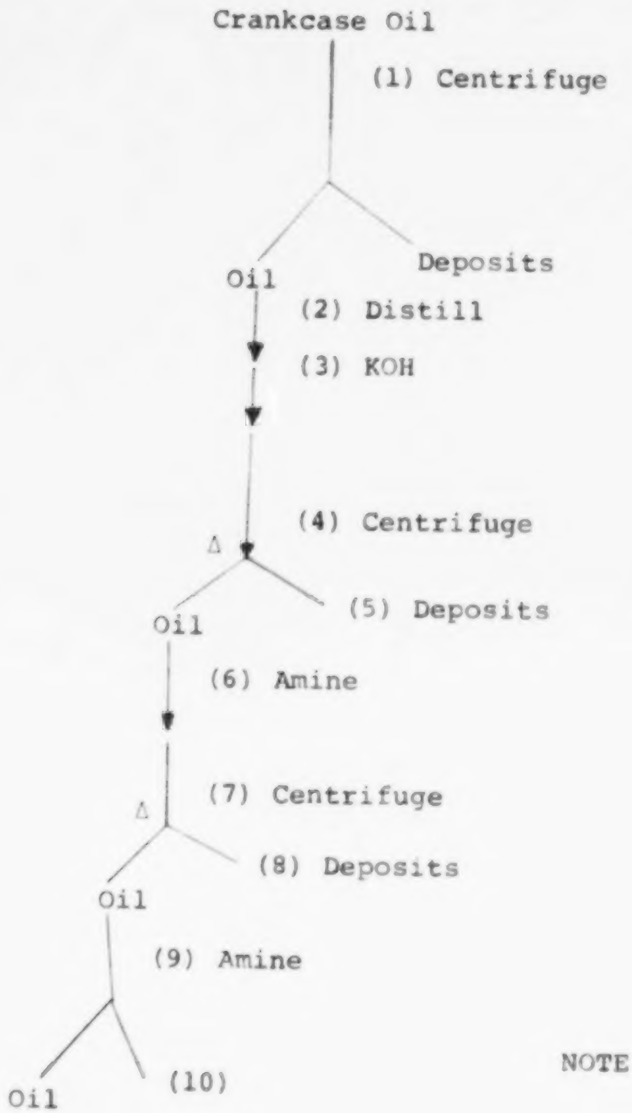
Procedure:

1. Centrifugation @ 31,890 G for 30 minutes to remove the preformed solids.
2. Vacuum distillation of the desludged oil to remove light ends and water. The terminal temperature at the still was 431°F and the vapor temperature was 392°F.
3. To obviate reaction of the amine with acids in the used oil, a 10 weight percent solution of 30% pottassium hydroxide was added to each sample that was treated. The mixture was then heated with agitation for 1 hour @ 150°F. (Each experiment involved two samples.)
4. The oil was again centrifuged @ 10,000 **G** for 30 minutes.
5. The residue was carefully washed and the percentage recorded.
6. Variable amounts of DET were added to the supernatant oil, and heated for one hour @ 150°F.
7. The oil was again centrifuged at 31,890 G for 30 minutes.
8. The percent residue was determined.
9. An equal percentage of DET was again added with agitation for one hour at 150°F.
10. The oil was centrifuged at 31,890 G for 30 minutes.
11. The percent residue was determined.

The accompanying Figure 4 illustrates the procedure followed.

Table 32. TREATMENT OF CRANKCASE OIL WITH DIETHYLENETRIAMINE (DET) (Continued)

	Experiment No. 1	Experiment No. 2	Experiment No. 3
Additive	15% DET	Heat Only	5% DET
Residue after KOH Treatment (Step 5)	0.58%, 0.68%	No KOH Treat. .56%, .59%	2.2%, 2.1%
Residue after Additive Treat. (Step 8)	1.11%, 1.27%	.346%, .343%	1.9%, 3.2%
Residue after Additive Treat.2 (Step 11)	0.15%, 0.16%	.22%, .21%	0.27%, 0.50%
Total Deposits	1.83%, 2.11%	1.13%, 1.15%	4.4%, 5.8%


Crankcase Oil
(1) Centrifuge
Oil
Deposits
(2) Distill
(3) KOH
(4) Centrifuge
Δ
Oil
(5) Deposits
(6) Amine
(7) Centrifuge
Δ
Oil
(8) Deposits
(9) Amine
Oil
(10)

NOTE: See Table 32 for results

TREATMENT OF CRANKCASE
OIL WITH DET-EXPERIMENTAL
PROCEDURE

FIGURE 4

Table 33. LEAD RECOVERY FROM CRANKCASE WASTE OILS

1.	NORCO tank bottoms (green)+	42.92% Pb*
2.	Sludge obtained by centrifugation (Centrico bowl type) of crankcase waste oil	13.52% Pb
3.	Sludge obtained by centrifugation (Centrico bowl type) of crankcase waste oil treated with 2 parts of butanol/part oil	15.88% Pb

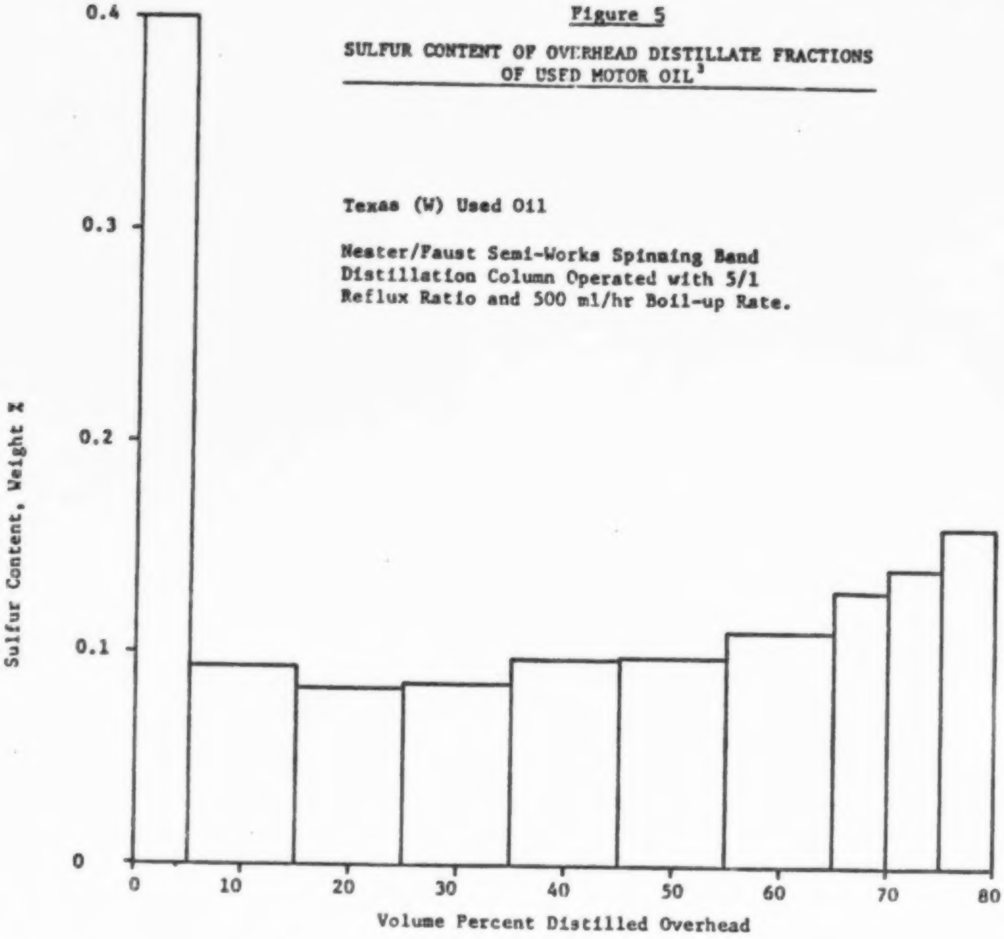
---

* By semi-quantitative spectrographic analysis 10-100%--Pb; 1-10%--Ca; 0.1-1.0%--Mg, Fe, Al, Cw, Si, Zn, Ba; 0.01-0.1%--Cr, Sn, Ni, V, Mo, Mn, Ti; 0.001-0.01%--B, Bi, Ag, Sr; Not Detected--P, Sb, As, Nb, Be, Zr, Cd, In, Na, Co, K, Hg

+ Bottoms from tank holding vacuum distillation bottoms fraction.

Figure 5

SULFUR CONTENT OF OVERHEAD DISTILLATE FRACTIONS OF USED MOTOR OIL[3]



Texas (W) Used Oil

Nester/Faust Semi-Works Spinning Band Distillation Column Operated with 5/1 Reflux Ratio and 500 ml/hr Boil-up Rate.

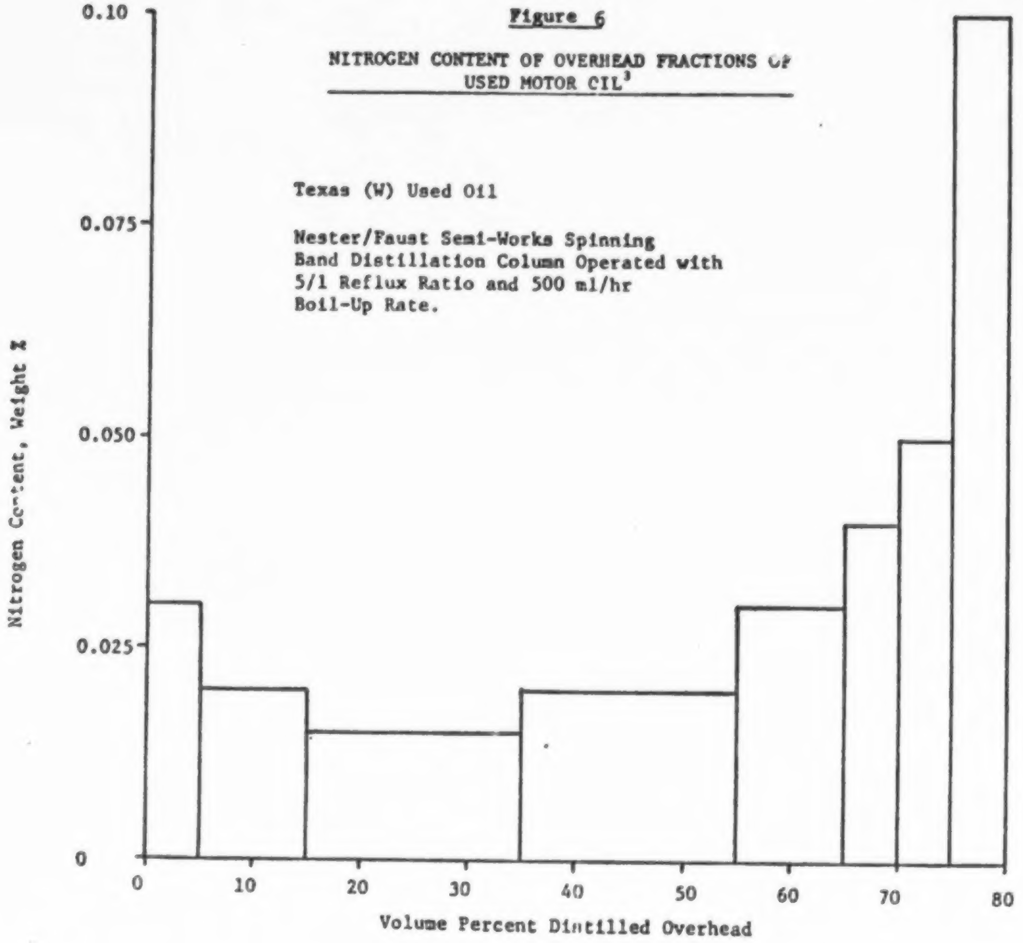


Figure 6

NITROGEN CONTENT OF OVERHEAD FRACTIONS OF USED MOTOR OIL[3]

Texas (W) Used Oil

Nester/Faust Semi-Works Spinning Band Distillation Column Operated with 5/1 Reflux Ratio and 500 ml/hr Boil-Up Rate.

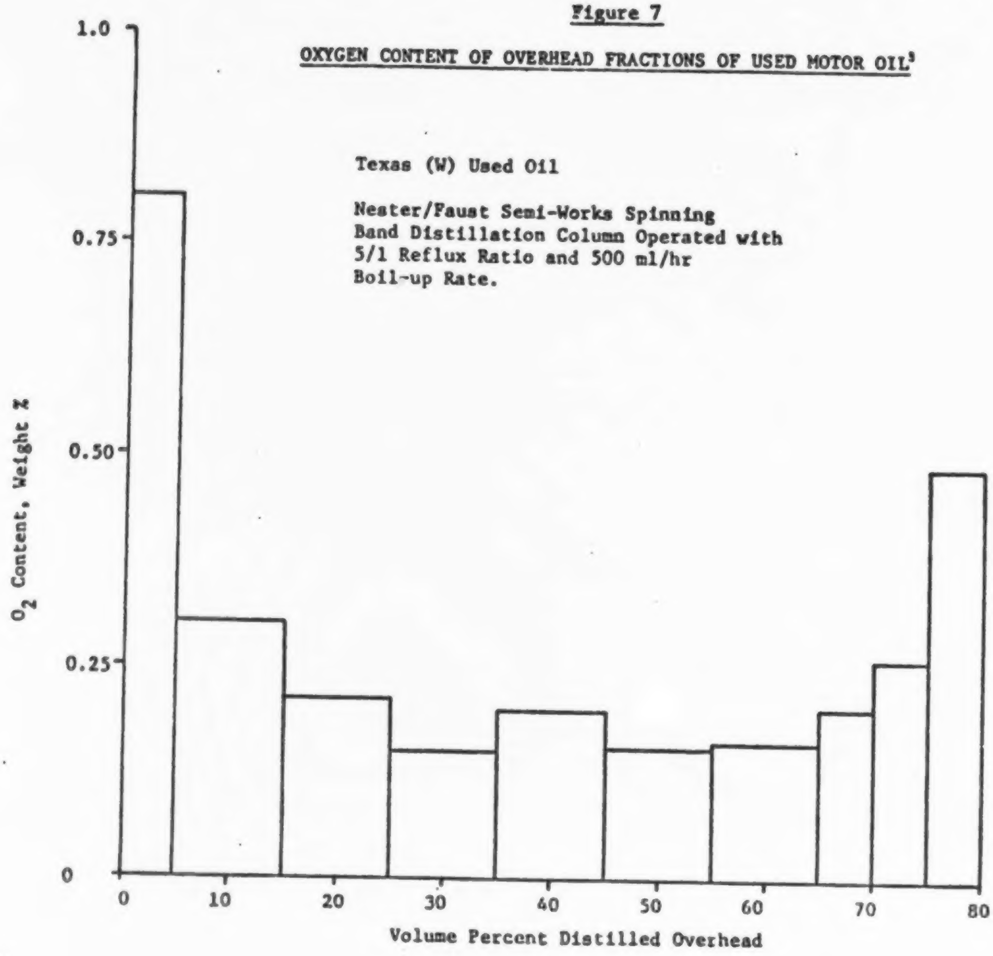


Figure 7

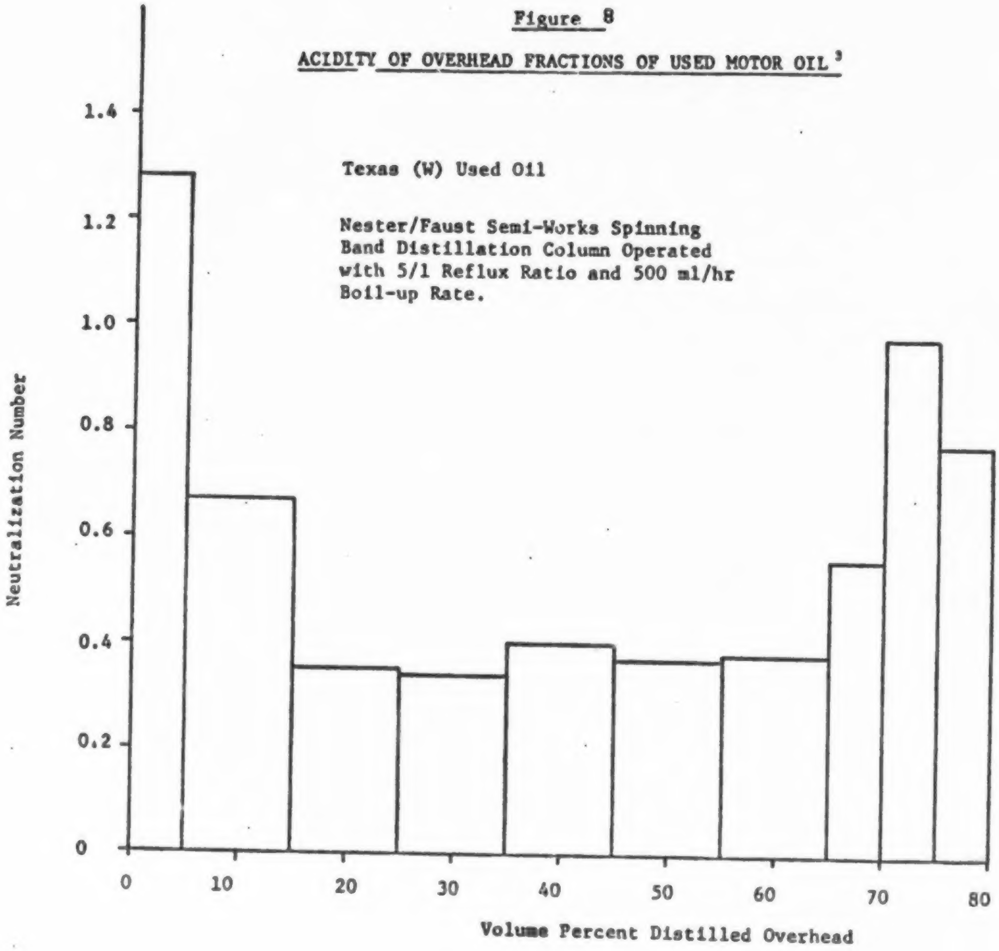
OXYGEN CONTENT OF OVERHEAD FRACTIONS OF USED MOTOR OIL[3]

Texas (W) Used Oil

Nester/Faust Semi-Works Spinning Band Distillation Column Operated with 5/1 Reflux Ratio and 500 ml/hr Boil-up Rate.

Figure 8

ACIDITY OF OVERHEAD FRACTIONS OF USED MOTOR OIL[3]



Texas (W) Used Oil

Nester/Faust Semi-Works Spinning Band Distillation Column Operated with 5/1 Reflux Ratio and 500 ml/hr Boil-up Rate.

This fact led to investigation of the possibility of lead recovery. Two major avenues were investigated. In the first, the lead was further concentrated into a solid material/ in the second the bottoms fraction was prepared for introduction into secondary lead smelting as a fuel.

Lead Concentration

Lead in the bottoms fraction can be concentrated by treating with naphtha to precipitate the solids, followed by evaporation of volatile hydrocarbons from the precipitated solids. Supporting data are shown in Tables 34-36.

Solid material containing 32.6% lead, prepared as shown in Table 35, was sent to the American Smelting and Refining Company for laboratory evaluation. The evaluation showed that, although lead recovery from this fraction was feasible, special processing schemes would have to be developed to handle it because of the hydrocarbons present.

Similar work and solvent treating tests were conducted on other lead-containing fractions, and on heavy bottoms material recovered from the Schuylkill River following a hurricane (Berk's bottoms). These are reported in Tables 37-39.

Use of Bottoms in Secondary Lead Smelting

At the end of 1971, there were 23 firms operating approximately 45 secondary lead smelting plants in the U.S.[4] Reverberatory, blast, and pot furnaces are commonly used in these plants. Both reverberatory and blast furnaces must be protected by high efficiency air pollution control equipment to minimize particulate (and lead) emissions to the atmosphere. In the reverberatory furnace, a large refractory chamber, lead scrap material is fed and melted by firing burners directly into the chamber. In the blast furnace (similar to the blast furnace used in iron making) lead scrap is mixed with coke, which acts both as a fuel and as a reductant, and fed to the top of the furnace. Air is introduced at the furnace tuyeres. Molten slag and lead are removed from the base of the furnace.

Table 34. CENTRIFUGATION OF CRANKCASE OIL BOTTOMS

1:1 Wt Ratio Naphtha/Bottoms
5000 G for 1 Hour

Charge:*	Bottoms from CCO Bottoms Tank	CCO Bottoms	CCO Bottoms(8°API)
Oil Layer, ml/g$^+$	1.5	2.2	1.99
Water Layer, ml/g$^+$	0.1	-	-
Residue, wt. %$^+$	21.3	5.6	12.1
%Pb in Charge	3.90	1.90	-
% Pb in Oil Layer	0.22	-	-
% Pb in Residue	20.3	24.1	-
% C in Residue	15.5	29.8	-
Other Metals in Residue			
1-10%	Si,Pb,Zn,Ca,Ba	Pb,Zn,Ca,Ba	- -
0.1-1%	Al,Fe,Cd,Mg,P,Cu,Ti	Al,Fe,Si,Mg,P,Cu	-
0.01-0.1%	Ni,Cr,Sn,Mn,B,Mo	Ni,Cr,Cd,Sn,Mn,B,Mo,Ti	
0.001-0.01%	Bi,Ga,Sb,V,Ag,Zr,Co,Pt,Tl	Bi,Sb,V,In,Co,Pt,Tl	
<0.001%	In,Be	In,Ag,Zr	
Not detected	Ge,As,Hg,Au,Na,W	Ge,Ga,As,Hg,Au,Be,W	

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* CCO = crankcase oil
CCO bottoms = bottoms from vacuum distillation

$^+$ Based on weight of bottoms

Table 35. "DRY LEAD ORE" FROM BOTTOMS OF CRANKCASE OIL BOTTOMS TANK

Procedure: Mixture of 3 parts of naphtha to one part of bottoms settled for two weeks; supernatant decanted; solids pentane washed and dried on a room radiator.

Product: Solids dry to touch
32.6 % Pb
31.5 % C
1-10 % Si, Zn, Ca, Ba
25-40 % volatile at 400°F (avg. 32 %)

Table 36. RESIDUE SEPARATION FROM NORCO CRANKCASE OIL TANK BOTTOMS BY NAPHTHA DILUTION AND SETTLING

CHARGE: 300 gallons of bottoms from crankcase oil tank

PROCEDURE: Mixture of 3 parts NORCO naphtha to 1 part bottoms allowed to settle for 14 days. Supernatant liquid decanted leaving 180 lbs. of solids (approx. 7.2 wt. %)

PRODUCT: Volatiles at 220°F (overnight) - 26.9%

Lead content - 11.7 wt. %
Carbon content - 38 wt. %

Table 37. SOLIDS CONTENT OF CRANKCASE OIL BOTTOMS

	NORCO Bottoms*	Berks Bottoms$^+$
Charge:	10.0495 g. oil 10.4865 g. pentane	10.3864 g. oil 10.4642 g. pentane
Result:	After centrifuging 30 min. at 32,000 G 1.7602 g. solids (17.51 % solids)	2.3390 g. solids (22.54 % solids)
Charge:	179.5 g. oil 179.5 g. pentane	
Result:	After one month settling 50.0 g. residue (wet with pentane) (27.85% solids)	

* NORCO settled bottoms - 0.9725 s.g.
$^+$ Berks aged bottoms - 1.0507 s.g.

Table 38. UPGRADING BERK'S BOTTOMS (FROM SPILL) BY NAPHTHA DILUTION AND FILTRATION

	As Recovered	After 1:1 dilution with 43° API naphtha and filtration through Buchner Funnel
Water	47.6	2.3
Ash on Ignition	13.5	1.0
Combustibles	80.2	99.
Cu	0.060	0.008
Ba	1.1	0.13
Ca	0.40	0.08
Cr	0.004	0.001
Al	0.31	0.01
Fe	0.57	0.07
Mg	0.06	0.01
Mn	0.02	0.002
Na	0.42	0.05
Ni	0.006	0.001
P	0.34	0.09
Pb	3.8	0.06
Si	2.3	0.04
Sn	0.008	0.002
Ti	0.008	0.001
Zn	0.21	0.04

Table 39. SOLVENT TREATMENT OF BERK'S BOTTOMS

Solvent	Treat ml oil/ ml Solvent	Results			
		1 hr		5 days	
		ml oil	Solvent	ml oil	Solvent
Amyl Alchol/ 10% P	5/5	5.5	Cloudy	5.0	Clear/ Dark Yellow
Amyl Alcohol	5/5	5.0	Good Septn.	5.0	
Amyl Alcohol/ 10% P	4/8	5.4	Good Septn.	4.5	
Amyl Alcohol/ 10% P	2/8	4.4	Good Septn.	4.2	
Methanol/10% P	5/5	-	Sl. Cloudy	-	Coagulated
Methanol/10% P	4/8	7.5	Cloudy Yellow	7.5	Clear
Methanol/10% P	2/8	-	Coagulated		Coagulated
50% Methanol/ 50% P	2/8	-	Coagulated	7.1	Coagulated
Isopropanol/ 10% P	5/5	6.5	Sl. Cloudy	6.5	Sl. Yellow
Isopropanol/ 10% P	4/8	6.5	Sl. Cloudy	6.5	Sl. Yellow
Isopropanol/ 10% P	2/8	5.5	Sl. Cloudy	5.5	Light Yellow
Amyl Alcohol/ 10% P	5/5	6.5	Good Septn.	6.5	Good Septn.
Amyl Alcohol/ 10% P	2/8	2.5	Good Septn.	2.2	Good Septn.
50% Butane Diol/ 50% MEK	5/5		Coagulated	8.8	Coagulated

P = Pentane

Based on preliminary tests conducted by NL Industries, and an analysis of lead smelter operations, it is felt that the most promising method of using the crankcase waste oil bottoms in secondary lead smelters is to replace or partially replace the fuel normally fired in the reverberatory furnace. In this way, both fuel and lead values could be realized, with the lead contained in the bottoms captured either in the furnace or in the baghouse used for air pollution control. The baghouse material is normally recycled for lead recovery.

After preliminary tests showed that combustion of the bottoms was feasible, a decision was made to conduct a full scale test on a reverberatory furnace in an NL Industries plant. The work was done under a grant from the U. S. Environmental Protection Agency. The results will be reported in the near future in a separate document.

CATALYTIC HYDROGEN TREATMENT

Hydrogen is commonly used as a reagent in conventional petroleum refining to remove sulfur and nitrogen from petroleum fractions. The finishing of lube stocks with hydrogen has largely replaced acid and clay treatment. Hydrogen treatment is usually conducted at 300 to 1000 psi and 500 to 700°F over catalysts containing a cobalt/molybdenum or nickel/molybdenum complex.

In this and other work,[3] it has been shown that catalytic hydrogen treatment can be used to upgrade distillate fractions obtained by vacuum distillation of crankcase waste oil. Nitrogen and oxygen is removed from the distillate fraction, forming NH_3 and H_2O. Some sulfur may also be removed from the distillate, which is already low in sulfur, as H_2S. These impurities are oxidized or scrubbed from a purge gas stream. The purification process improves the stability, color, and odor of lube distillates, as shown in Table 40.

Table 40. PRODUCT QUALITY IMPROVEMENT BY CATALYTIC HYDROTREATMENT OF CRANKCASE OIL DISTILLATE

	Waste Crankcase Oil	Untreated Distillate	Hydro-Treated Distillate
HRI Data*			
ASTM Color	D8	L 7.5	L 2.5-L 3.5
Odor	Offensive	Offensive	Odor Removed
Exxon Data[3]			
ASTM Color	Black	Black	Lt. 1.0-1.5
Neutr. No.	5.87	0.51	0
Con. Carbon, Wt.%	3.33	0.01	0.001
Sulfur, Wt.%	0.30	0.12	0.1-0.05
Nitrogen, Wt.%	0.08	0.018	0.002-0.006

* See Appendix E

The results of small scale pilot plant runs on NORCO distillate are provided in Appendix E. The type of experiment conducted does not answer two other important questions about hydrogen treatment, namely hydrogen consumption and catalyst life. Hydrogen consumption was estimated to be 70 to 160 standard cubic feet per barrel of distillate by hydrogenation bomb tests and by changes in physical and chemical properties. The basis for these estimates is provided in Appendix E.

No tests were made to determine catalyst life. However, neither the catalytic hydrogen treatment tests run in this work (67 hours), or the other work previously mentioned[3] (about 100 hours) showed any sign of catalyst deactivation.

CHEMICAL REDUCTIONS

The use of hydrides for the reduction of precursors and color and odor producing compounds in lube distillates may be a practical alternative to catalytic hydrotreating. Reductants studied in this work were lithium aluminum hydride, sodium and potassium borohydride, and sodium aluminum diethyl dihydride. Other aluminum hydrides, borohydrides, and similar compounds might also be considered.

Sodium borohydride rapidly reduces most aldehydes, peroxides, and hydroperoxides. Most ketones are reduced at a much slower rate than aldehydes. Carboxylic acids, carboxylic acid esters, amides, nitriles, and nitro compounds are not normally reduced. Other types of compounds which are usually reduced by sodium borohydride are: acid chlorides, aromatic azides, organic disulfides, carbon/carbon and carbon/nitrogen double bonds, lactones, ozonides, cyclic quarternary ammonium salts, and Schiff bases. Reductive N-alkylations, cleavages, cyclizations, deaminations, and deoxygenations can occur. Numerous inorganic reductions are also possible.[5,6] Technical information on sodium borohydride is provided in Appendix L. Qualitative infrared information confirmed that the borohydrides used did reduce nitrogen oxide compounds (6.3 microns), but few carbonyls (5.95 microns).

Aldehyde, ketone, organic acid, ester, organic acid chloride, oxime, amide, nitrile, and nitro compound reductions by sodium aluminum diethyl dihydride (OMH-1) are known.[7] The ability to reduce organic acids, esters, amides, nitriles, and nitro compounds distinguishes OMH-1 from sodium borohydride, making it possibly more attractive for waste oil treatment. However, OMH-1 must be handled under an inert atmosphere. Technical information is provided in Appendix L. Qualitative infrared information confirmed the reactivity of OMH-1 for nitrogen oxide and carbonyl compounds.

Sodium borohydride can in theory generate 4 moles of H_2 per mole by hydrolysis:

$$NaBH_4 + 4\ H_2O = NaB(OH)_4 + 4\ H_2.$$

Therefore, one pound of $NaBH_4$ can generate 37.94 standard cubic feet (32°F, 1 atm) of hydrogen. Correspondingly, one pound of sodium aluminum diethyl dihydride can theoretically generate 13.04 standard cubic feet of hydrogen by hydrolysis:

$$NaAl(C_2H_5)_2H_2 + 4\ H_2O = NaAlO_2 + 2C_2H_5OH + 4\ H_2.$$

The potential usefulness of chemical reduction reagents depends upon selectivity for compounds which contribute to poor lube properties, the cost of the reagent, and additional steps required, for example to eliminate odors and remove precipitated salts. The odor problem will be discussed further. A haze which appeared in treated samples due to salts, water (where present), and other reaction products can be removed by washing (e.g. amines), redistillation, and/or filtering.

Redistillation of a blend of NORCO No. 3 and 4 distillate yields fractions showing an ASTM color of 2.4 to 4.2, with most of the light cuts at 2.4 (Table 41). Addition of solid KBH_4 or $NaBH_4$ to the distillation flask can improve the color to as low as 1.1 to 1.75, depending on the concentration (Tables 41 to 47). It appears that at least 0.016 weight percent KBH_4 was required to make a significant improvement in color. However, odor was not improved in any case. Some degradation of color after several weeks was noted in sample bottles containing borohydride treated oils.

Table 41. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH AND WITHOUT POTASSIUM BOROHYDRIDE

Distillation of NORCO #3 and #4 blend and .08 weight % KBH_4

% Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum (Torr)	ASTM Color
10	580	260	49	1.75
20	600	280	58	1.75
30	612	295	49	1.75
40	630	304	49	1.75
50	640	310	47	1.75
60	658	328	47	1.75
70	676	332	47	2.40
80	700	352	47	2.80
90	730	358	47	4.20

Distillate recovery - 92%

Distillation of NORCO #3 and #4 blend - no KBH_4 added

10	580	255	48	2.4
20	600	280	48	2.4
30	618	295	48	2.4
40	636	305	49	2.4
50	648	315	49	2.4
60	665	320	49	2.8
70	674	328	47	2.8
80	692	334	47	3.5
90	730	348	47	4.2

Recovery - 93.5%

Distillation of NORCO #3 and #4 blend and 0.016 weight % KBH_4

10	405	190	57	1.75
20	524	195	49	1.75
30	570	242	48	1.75
40	590	268	48	1.75
50	610	270	48	1.75
60	630	275	48	2.75
70	677	278	48	2.75
80	700	285	48	3.50
90	738	270	48	7

Table 42. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH 0.0016% POTASSIUM BOROHYDRIDE*

Distillation of NORCO #3 and #4 blend and 0.0016 weight % KBH_4

% Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum (Torr)	ASTM Color
10	538 (575)	240 (280)	51 (86)	2-3/4 (2-3/8)
20	548 (615)	242 (305	51 (77)	2-3/4 (2-3/4)
30	562 (640)	248 (318)	51 (73)	2-3/4 (2-3/8)
40	576 (660)	250 (330)	51 (73)	2-3/4 (2-3/8)+
50	595 (683)	254 (343)	51 (75)	2-3/4 (2-3/8)
60	610 (702)	257 (350)	51 (77)	3-1/2 (2-3/4)
70	657 (720)	260 (360)	51 (92)	3-1/2 (3 1/2)
80	672 (765)	280 (375)	51 (77)	4-1/8 (4-3/4)
90	704	270	51	4-3/4

* Numbers in parentheses represent a second experiment

+ Original Color as above = 2-3/8
Color after 12 days = 3-1/2
Color after heat treatment with agitation at 200°F for 45 minutes = 3-1/2

Table 43. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH 1.25% POTASSIUM BOROHYDRIDE

Distillation of NORCO 3 and 4 blend* with 1.25 weight % KBH_4

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum (Torr)	ASTM Color
initial	410	75	48	
10	560	235	43	1.75
20	588	270	43	1.1
30	600	278	43	1.1
40	614	282	43	1.1
50	630	290	43	1.1
60	651	298	43	1.1
70	673	303	43	1.75

A semi-quantitive reduction of the 1st sample was shown by infra-red analysis.

* 56.2% No. 3 and 43.8% No. 4 by weight

Table 44. DOUBLE DISTILLATION WITH SODIUM BOROHYDRIDE TO IMPROVE COLOR

TREATMENT: 0.05% $NaBH_4$ added directly to distillation flask with NORCO No. 3 and 4 blend

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum (Torr)	ASTM Color
10	575	268	55	1-3/4
20	600	280	55	1-3/4
30	628	295	55	1-3/4
40	638	305	55	1-3/4
50	648	325	55	1-3/4
60	668	335	55	2-3/4
70	695	340	55	2-3/4
80	740	335	55	4-3/4
85	750	-	55	--

TREATMENT: Redistillation of above cuts above 50% with additional 0.05% $NaBH_4$ (mixture color = 3+)*

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum (Torr)	ASTM Color
10	594	260	50	2-3/8
20	607	273	55	1-3/4
30	618	276	55	1-3/4
40	627	278	55	1-3/4
50	642	290	55	1-3/4
60	660	303	55	1-3/4
70	690	310	55	2-3/4
80	720	315	55	3-1/2
85	-	-	-	--

* About 1/3 of charge was a cut from a previous run with a 2-7/8 ASTM color

Table 45. REDISTILLATION OF NORCO NO. 3 (LIGHT SIDE-STREAM) CRANKCASE OIL DISTILLATE WITH 0.001% SODIUM BOROHYDRIDE

TREATMENT: 0.0017 grams of $NaBH_4$ added to 200 ml NORCO No. 3 followed by distillation

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum, Torr	ASTM Color
Initial Boiling Point	370	75	82	-
10	523	180	80	1 3/4
20	553	205	80	1 3/4
30	576	223	80	1 3/4
40	585	233	80	2 3/8
50	586	240	80	2 3/4
60	605	260	80	3 1/2
70	660	210	80	4 3/4

Table 46. REDISTILLATION OF NORCO NO. 4 (HEAVY SIDESTREAM) CRANKCASE OIL DISTILLATE WITH 0.001% SODIUM BOROHYDRIDE

TREATMENT: NONE

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum, Torr	ASTM Color
Initial Boiling Point	494	90	60	-
10	543	220	65	2 3/4
20	558	230	65	2 3/4
30	575	243	65	2 3/4
40	588	250	65	3 1/2
50	613	255	65	3 1/2
60	645	250	65	4 1/8
70	688	230	65	4 3/4
TREATMENT: 0.0017 grams of $NaBH_4$ added to 200 ml. NORCO No. 4 followed by distillation with stirrer on and N_2 purge				
Initial Boiling Point	540	143	55	-
10	559	236	55	2 3/4
20	575	250	55	2 3/4
30	593	258	55	2 3/4
40	645	267	55	3
50	657	273	55	3 1/2
60	671	277	55	4 1/8
70	690	281	55	4 1/8
80	710	273	55	5 1/2

Table 47. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH 0.016% POTASSIUM BOROHYDRIDE AND 0.156% ALUMINUM CHLORIDE

TREATMENT: 0.0272 grams of KBH_4 and 0.272 grams of $AlCl_3$ added to blend of NORCO No. 3 and 4 followed by distillation with stirrer on and N_2 purge

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum, Torr	ASTM Color
10	510	195	49	1 3/4
20	546	220	50	1 3/4
30	570	230	50	1 3/4
40	590	235	51	2 3/8
50	613	247	45	2 3/8
60	638	273	43	2 3/4
70	650	283	43	2 3/4

Results obtained using a soluble water solution of 12 weight percent $NaBH_4$ in 40% NaOH solution were similar, but substantial color improvement was indicated at treatments as low as 0.0016 weight percent $NaBH_4$ (Tables 48-54). Again, no odor improvement was noted.

About 0.01 weight percent $NaBH_4$ seemed to be required for color improvement when using an agent containing 5 weight percent $NaBH_4$ in oil (Tables 55-59). Odor was not improved.

It was found that odor improvement was possible by treating the distillate with hot water or potassium hydroxide solution (Tables 60-63), but color was not improved. Unfortunately, the odor reappears again after redistillation (Tables 62 and 63). This occurred even when Solvent 150 Neutral was distilled.

A single experiment with 0.016 weight percent lithium aluminum hydride treatment showed little or no color or odor improvement (Table 64), indicating little incentive to pursue this approach.

The use of sodium aluminum diethyl dihydride (OMH-1) not only improves color, but reduces odor to some extent, probably by the elimination of carbonyl as noted by infrared. (Tables 65-71). However, as previously indicated, odor does return when the oils are reheated for distillation.

When treating distillation cuts with OMH-1 after sodium borohydride treatment and distillation, all samples got cloudy with precipitation of some black material (pyridine odor).

Several experiments with borohydride treatment of raw crankcase oil indicated some possibility of this approach being successful, but distillation would probably be necessary to remove the darker color heavy cuts, even if centrifugation could be used to remove metals and sludge (Tables 72-76).

Laboratory prepared bottoms also showed some improvement in color when treated with potassium borohydride (Table 77).

The relatively low quantity of hydride reagents required to improve color and odor, and the mild conditions used, suggest that the impurities are reactive and that cayalytic hydrotreating could possibly be improved by the development of more selective and active catalysts.

Table 48. EFFECT OF TEMPERATURE ON TREATMENT OF CRANKCASE OIL DISTILLATE WITH 0.12% SODIUM BOROHYDRIDE

TREATMENT: Blend of NORCO No. 3 and 4 treated with 1% SWS (soluble water solution containing 12% $NaBH_4$ in 40% NaOH water solution) at 180°F for 1 hour followed by centrifugation at 32,000 G for 30 minutes. The supernatant was distilled as follows.

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum (Torr)	ASTM Color
10	550	225	56	1-3/4
20	558	227	53	1-3/4
30	580	237	54	1-3/4
40	606	247	55	1-3/4
50	640	257	55	1-3/4
60	675	265	55	2-3/8
70	688	283	55	2-3/4
80	738	250	55	4-3/4

TREATMENT: 1% SWS added directly to distillation flask with No. 3 and 4 blend.

10	545	187	55	1-1/8
20	550	232	55	1-1/8
30	560	237	55	1-1/8
40	584	247	55	1-1/8
50	615	255	55	1-1/8
60	660	260	55	1-1/8
70	688	270	55	1-3/4
80	740	260	55	2-3/4

Table 49. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH SODIUM BOROHYDRIDE -- EFFECT OF CONCENTRATION*

Vol. % Recovered	Still Temperature, °F						Vapor Temperature, °C					
	0	0.0008	0.0016	0.012	0.024	0.071	0	0.0008	0.0016	0.012	0.024	0.071
10	595	578	550	565	578	585	270	242	250	247	260	257
20	602	590	590	595	600	610	276	260	260	267	268	263
30	615	608	610	608	618	615	278	265	265	270	275	265
40	630	620	630	621	630	625	280	270	268	272	285	270
50	634	639	648	665	650	650	283	272	270	282	287	258
60	660	648	665	675	662	660	285	275	275	288	290	278
70	678	698	685	685	688	670	287	285	280	290	292	284
80	704	740	715	710	715	702	287	280	283	292	292	285
90	765	-	760	770	-	738	295	-	280	288	-	268

	Vacuum, Torr						ASTM Color					
10	50	55	60	55	55	55	2 3/4	.2 3/4	1 3/4	1 3/4	1 3/4	1 1/8
20	55	60	60	55	55	55	2 3/4	2 3/8	1 3/4	1 3/4	1 3/4	1 1/8
30	55	60	60	55	55	55	2 3/4	2 3/8	1 3/4	1 3/4	1 3/4	1 1/8
40	55	60	60	55	55	55	2 3/4	2 3/8	1 3/4	1 3/4	1 3/4	1 1/8
50	55	60	60	55	55	55	2 3/4	2 3/8	2 3/4	1 3/4	1 3/4	1 1/8
60	55	60	60	55	55	55	2 3/4	2 3/4+	2 3/4	2 3/8	2 3/8	1 1/8
70	55	60	60	55	55	55	3 1/2	2 3/4	2 3/4	2 3/4	2 3/8	1 3/4
80	55	60	60	55	55	55	4 1/4	3 1/2	3 1/2	3 1/2	3 1/2	1 3/4
90	55	60	60	55	55	55	6 1/2	--	6 1/2	--	--	--

* Blend of NORCO No. 3 and 4 treated with 0, 0.0008, 0.0016, 0.012, 0.024, or 0.071 NaBH$_4$ as a 12%NaBH$_4$/40%NaOH water solution.
+ After this cut an additional 0.0008%NaBH$_4$ added (vacuum broken).

Table 50. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH 0.012% SODIUM BOROHYDRIDE AFTER WASHING WITH POTASSIUM HYDROXIDE

TREATMENT: Wash 200 ml. of NORCO No. 3 and 4 blend with 5 vol.% of 10 wt.% KOH solution, centrifuge at 3000 G for 30 min. Add 0.175 grams of 12% NaBH$_4$/40% NaOH water solution and distill.

% Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum, Torr	ASTM Color
Initial Boiling Point	430	60	60	-
10	540	233	60	1 3/4
20	557	250	60	1 1/8
30	565	260	60	1 1/8
40	585	265	60	1 1/8
50	597	270	60	1 1/8
60	610	275	60	1 3/4
70	632	278	60	2 3/8
80	685	290	60	2 3/4
90	760	250	60	5 3/4

Table 51. PRETREATMENT OF CRANKCASE OIL DISTILLATE WITH 10% H_2SO_4 PRIOR TO 0.024% BOROHYDRIDE TREATMENT

TREATMENT: Wash 200 ml. of blend of NORCO No. 3 and 4 with 5 vol. % of 10 wt. % H_2SO_4, centrifuge at 3000 G for 30 min., and repeat. Add 0.024 wt. % $NaBH_4$* and redistill.

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum Torr	ASTM Color
Initial	456	58	55	-
10	545	245	55	1 3/4
20	568	260	55	1 1/8
30	585	270	55	1 1/8
40	602	280	55	1 1/8
50	615	285	55	1 1/8
60	630	288	55	1 3/4
70	643	292	55	1 3/4
80	670	300	55	1 3/4
90	710	313	55	3 1/2

No improvement in odor noted.

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* As 12 wt. % $NaBH_4$ in 40 wt. % NaOH water solution.

Table 52. PRETREATMENT OF CRANKCASE OIL DISTILLATE WITH 5% KOH FOLLOWED BY 10% H_2SO_4 PRIOR TO 0.012% BOROHYDRIDE TREATMENT

TREATMENT: Wash 200 ml. of blend of NORCO No. 3 and 4 twice with 10 vol. % of 5 wt. % KOH and then twice with 5 vol. % of 10 wt. % H_2SO_4. Add 0.012 wt. % $NaBH_4$* and redistill.

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum Torr	ASTM Color
Initial	480	190	55	-
10	558	260	55	1 1/8
20	576	275	55	1 1/8
30	591	285	55	1 1/8
40	606	288	55	1 3/4
50	620	290	55	1 3/4
60	627	295	55	1 3/4
70	646	296	55	1 3/4
80	660	298	55	2 3/4
90	706	305	55	3 1/2

No improvement in odor noted.

* As 12 wt. % $NaBH_4$ in 40 wt. % NaOH water solution.

Table 53. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH 0.0012% SODIUM BOROHYDRIDE*

Pretreatment: A. Add 16.5 grams stainless metal sponge to 200 ml oil.

B. None

C.1) Heat oil to 400°F with agitation
2) Add borohydride
3) Hold at 400°F for 6 minutes
4) Quench flask
5) Centrifuge @ 31,000 G for 30 minutes

Vol. % Recovered	Still Temp., °F			Vapor Temp., °C			Vacuum, Torr			ASTM Color		
	A	B	C	A	B	C	A	B	C	A	B	C
10	538	550	570	217	250	223	65	60	55	1 3/4	1 3/4	1 3/4
20	552	590	588	238	260	238	65	60	55	1 3/4	1 3/4	1 3/4
30	570	610	600	243	265	240	65	60	55	2 3/8	1 3/4	1 3/4
40	590	630	633	250	268	244	65	60	55	2 3/8	1 3/4	2 3/8
50	618	648	662	255	270	246	65	60	55	2 3/8	2 3/4	2 3/4
60	643	665	686	262	285	249	65	60	55	2 3/4	2 3/4	3 1/2
70	674	685	710	270	280	230	65	60	55	2 3/4	2 3/4	--
80	705	715	-	273	283	-	65	60	55	3 1/2	3 1/2	--
90	750	760	-	270	280	-	65	60	55	6 1/2	6 1/2	--

* Blend of NORCO No. 3 and 4 treated with 0.01% 12%$NaBH_4$/40%NaOH water solution.

Table 54. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH 0.012% SODIUM BOROHYDRIDE*

Pretreatment: A. None

B. Digestion at 150°F for 1 hour.

C. Digestion at 300°F for 1 hour followed by centrifugation at 32,000 G for 30 minutes.

Vol. % Recovered	Still Temp., °F			Vapor Temp., °C			Vacuum, Torr			ASTM Color		
	A	B	C	A	B	C	A	B	C	A	B	C
10	565	565	590	247	235	250	55	55	85	1 3/4	1 3/4	-
20	595	590	608	267	265	260	55	55	80	1 3/4	1 3/4	1 3/4
30	608	603	615	270	270	265	55	55	80	1 3/4	1 3/4	1 3/4
40	621	618	630	272	280	270	55	55	80	1 3/4	2 3/8	1 3/4
50	665	635	660	282	285	280	55	55	80	1 3/4	2 3/8	1 3/4
60	675	665	686	288	287	290	55	55	80	2 3/8	2 3/4	2 3/4
70	685	690	704	290	290	294	55	55	80	2 3/4	3 1/2	2 3/4
80	710	730	734	292	305	298	55	55	80	3 1/2	4 1/8	3 1/2
90	770	-	760	288	-	290	55	-	80	--	--	--

* Blend of NORCO No. 3 and 4 treated with 0.1% 12%$NaBH_4$/40%NaOH water solution.

Table 55. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH AN OIL/SODIUM BOROHYDRIDE MIXTURE*

TREATMENT: Blend of NORCO No. 3 and 4 charged to vacuum distillation with Borex.* Three tests equivalent to 0.005, 0.01, and 0.03 weight precent $NaBH_4$.

% Recovered	Still Temp., °F					Vapor Temp., °C				
	0.001	0.005	0.01	0.02	0.03	0.001	0.005	0.01	0.02	0.03
10	504	538	553	584	582	197	137	230	260	255
20	558	564	580	602	600	235	262	255	270	270
30	580	580	600	618	614	253	270	267	275	275
40	607	595	615	625	628	260	273	275	280	280
50	646	610	632	646	638	267	280	278	283	285
60	660	637	652	667	663	275	282	282	285	287
70	690	660	700	686	685	288	285	288	287	290
80	715	690	740	716	715	292	287	285	297	295
90	-	747	-	760	760+	-	270	-	270	270

% Recovered	Vacuum, Torr	ASTM Color				
		0.001	0.005	0.01	0.02	0.03
10	60	3 1/2	2 3/8	1 3/4	2 3/8	1 3/4
20	60	2 3/4	1 3/4	1 3/4	2 3/8	1 3/4
30	60	2 3/4	1 3/4	1 3/4	2 3/8	1 3/4
40	60	2 3/4	2 3/8	2 3/8	2 3/8	1 3/4
50	60	2 3/4	2 3/8	2 3/4	2 3/8	1 3/4
60	60	2 3/4	2 3/4	2 3/4	2 3/4	1 3/4
70	60	3 1/2	3 1/2	3 1/2	2 3/4	2 3/4
80	60	4 1/8	3 1/2	4 3/4	2 3/4	3 1/2
90	60	--	5 3/4	--	--	4 3/4

* Blend of NORCO No. 3 and 4 treated with 0.001, 0.005, 0.01, 0.02, and 0.03 wt.% $NaBH_4$ approximately 5 wt.% $NaBH_4$ in "Bayol 385" oil known as Borex (Ventron Corporation).

Table 56. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH SODIUM BOROHYDRIDE*

Vol. % Recovered	Still Temp., °F			Vapor Temp., °C			Vacuum Torr	ASTM Color		
	0.011	0.02+	0.024	0.011	0.02+	0.024		0.011	0.02+	0.024
Initial	440	450	425	150	58	40	55	-	-	-
10	510	545	535	215	235	225	55	1-3/4	1-3/4	1-3/4
20	532	560	560	250	260	253	55	1-3/4	1-3/4	1-3/4
30	555	570	570	257	265	258	55	2-3/8	1-3/4	1-3/4
40	570	580	583	265	268	260	55	2-3/8	1-3/4	1-3/4
50	590	600	590	270	270	262	55	2-3/8	1-3/4	1-3/4
60	607	618	598	275	274	264	55	2-3/8	2-3/8	1-3/4
70	645	657	605	280	273	268	55	3-1/2	3-1/2	2-3/4
80	668	700	650	285	268	255	55	3-1/2	4-3/4	3-1/2
90	750+	-	-	175	-	-	55	5-3/4	-	-

* Borex - 5 wt. % $NaBH_4$ in oil. Subheadings represent percent by weight $NaBH_4$ treatment.

+ 0.01% added initially and 0.01% added after the 40% cut was taken.

Table 57. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH 0.049% SODIUM BOROHYDRIDE*

Vol.% Recovered	Still Temp., ^{o}F	Vapor Temp., ^{o}C	Vacuum Torr	ASTM Color
Initial	500	135	55	-
10	553	255	55	2 3/4
20	570	275	55	1 3/4
30	587	280	55	1 3/4
40	600	290	55	1 3/4
50	608	295	55	1 3/4
60	618	297	55	1 3/4
70	642	299	55	2 3/4
80	676	290	55	3 1/2

* Borex-5 wt. % $NaBH_4$ in oil

Table 58. TREATMENT OF CRANKCASE OIL DISTILLATE WITH 0.01 WT. % SODIUM BOROHYDRIDE AT 600°F FOR SIX MINUTES

TREATMENT: Mixture of NORCO No. 3 and 4 distillate heated to 600°F, 0.01 wt. % $NaBH_4$ added,* mixture held at 600°F for 6 minutes, mixture quenched and then centrifuged at 31,000 G for 30 minutes to remove $NaBH_4$ from further reaction. Mixture then distilled.

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum Torr	ASTM Color
10	570	222	55	1 3/4
20	580	238	55	1 3/4
30	600	240	55	1 3/4
40	633	244	55	2 3/4
50	662	246	55	2 3/8
60	686	249	55	2 3/4
70+	710	230	55	3 1/2

* As Borex (5% $NaBH_4$ in oil).

+ Another 17% recovered in cold trap.

Table 59. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH 0.02% SODIUM BOROHYDRIDE AFTER WASHING WITH POTASSIUM HYDROXIDE

TREATMENT: Wash 200 ml. blend of NORCO No. 3 and 4 with 200 ml. of 50% KOH solution and centrifuge at 3000 G for 30 minutes. Add 0.7 grams of Borex (5% $NaBH_4$ in oil).

% Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum, Torr	ASTM Color
Initial Boiling Point	450	70	60	-
10	560	230	60	1 3/4
20	580	245	60	1 3/4
30	598	255	60	1 3/4
40	610	257	60	1 3/4
50	625	260	60	1 3/4
60	614	262	60	1 3/4
70	678	263	60	1 3/4
80	700	275	60	2 3/4
90	720	260	60	4 1/4

Table 60. DEODORIZATION OF NORCO BLENDED DISTILLATE WITH HOT WATER

TREATMENT: 150 ml. of NORCO No. 3 and 4 sidestream blend treated with 150 ml. of water at 200°F for 10 min.

RESULT: The odor in the oil phase was diminished, but not altogether gone. An odor appeared in the water phase. Phenol in water phase about 10 ppm.

Table 61. DEODORIZING CRANKCASE OIL DISTILLATE

Charge: 2nd 20 ml. cut (out of 200 ml. total) after 0.08% KBH_4 treat with distillation

Charcoal

150°F treatment -- some improvement but basic odor still present.

30% KOH

3 washes with 1 volume oil/volume KOH solution -- odor disappeared but oil cloudy after 20 hours standing.

10% $NaHCO_3$

1 wash with 1 volume oil/volume $NaHCO_3$ solution -- no odor improvement (cresylic-phenol type odor picked up)

Table 62. DEORDORIZATION OF NORCO BLENDED DISTILLATE WITH 10% KOH PRIOR TO BOROHYDRIDE TREATMENT

TREATMENT: Blend of NORCO No. 3 and 4 sidestream blend treated twice with 5 vol. % of a 10 wt. % KOH solution followed by centrifugation at 3000 G for 30 min.

RESULT: Odor removed. Water fraction contained odor.

TREATMENT: Redistillation of oil after KOH treatment with 0.012 % sodium borohydride (0.1 wt. % of 12 wt. % $NaBH_4$ in 40 wt. % aqueous sodium hydroxide solution).

Vol. % Recovered	Still Temp., ^oF	Vapor Temp., ^oC	Vacuum Torr	ASTM Color
initial	430	60	60	-
10	540	233	60	1 3/4
20	557	250	60	1 1/8
30	565	260	60	1 1/8
40	585	265	60	1 1/8
50	597	270	60	1 1/8
60	610	275	60	1 3/4
70	632	278	60	2 3/8
80	685	290	60	2 3/4
90	760	250	60	5 3/4

Odor reappears after distillation.

Table 63. ODOR REAPPEARANCE AFTER REDISTILLATION OF DEODORIZED/DECOLORIZED CRANKCASE OIL DISTILLATE

Charge: 30, 40, and 50% fractions of NORCO No. 3 and 4 blend redistilled with 0.016 wt.% sodium borohydride (ASTM color of 2 3/8 for each fraction). Typical crankcase oil distillate odor.

Treatment: Wash oil twice with 5 vol.% of 10 Wt.% KOH, centrifuge at 3000 G for 30 minutes, and distill.*

% Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum, Torr	ASTM Color
Initial Boiling Point	450	80	60	-
10	525	225	60	1 1/8
20	538	247	60	1 1/8
30	548	255	60	1 1/8
40	558	260	60	1 1/8
50	574	265	60	1 1/8
60	592	270	60	1 1/8
70	622	273	60	1 3/4
80	640	278	60	2 3/8
90	710	220	60	4 1/4

* Odor removed by KOH treatment, but reappears after distillation.

Table 64. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH 0.016% LITHIUM ALUMINUM HYDRIDE

TREATMENT: 0.0272 grams of $LiAlH_4$ stirred with 200 ml. NORCO No. 3 and 4 blend for 1 hour followed by distillation with stirrer on and N_2 purge.

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum, Torr	ASTM Color
10	507	200	57	2 3/4
20	535	215	57	2 3/8
30	548	230	57	2 3/8
40	563	237	57	2 3/8
50	567	238	57	2 3/4
60	590	240	57	2 3/4
70	615	215	57	3 1/2

Table 65. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH 0.26% OMH-1*

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum Torr	ASTM Color
Initial	490	65	55	-
10	558	247	55	1-1/8
20	575	265	55	1-1/8
30	593	270	55	1-1/8
40	605	273	55	1-1/8
50	618	277	55	1-3/4
60	635	280	55	1-3/4
70	650	282	55	1-3/4
80	655	284	55	2-3/4
90	695	280	55	4-3/4

* 0.26 wt.% OMH-1 as 26.3 wt.% OMH-1 (sodium aluminum diethyl dihydride) in toluene (containing 3-4% tetrahydrofuran).

Table 66. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH OMH-1*

Vol. % Recovered	Still Temp., ^{o}F	Vapor Temp., ^{o}C	Vacuum Torr	ASTM Color
Initial	480	160	55	-
10	522	210	55	2-3/8
20	550	235	55	1-3/4
30*	573	243	55	1-3/4
40	605	253	55	1-3/4
50*	620	258	55	1-3/4
60	638	259	55	1-3/4
70*	648	268	55	1-3/4
80	665	270	55	3-1/2
90	700	265	55	-

* 0.065 wt.T OMH-1 as 26.3 wt.% OMH-1 (sodium aluminum diethyl dihydride) in toluene (containing 3-4% tetrahydrofuran) added after each of the indicated cuts.

Table 67. REDISTILLATION OF CRANKCASE OIL DISTILLATE AFTER PRETREATMENT WITH OMH-1*

Vol. %	Still Temp., ^{o}F					Vapor Temp., ^{o}C					Vacuum	ASTM Color				
Recovered	0.026	0.052	0.078	0.13	0.26	0.026	0.052	0.078	0.13	0.26	Torr	0.026	0.052	0.078	0.13	0.26
Initial	420	460	487	520	425	40	45	145	195	64	55	-	-	-	-	-
10	520	552	520	554	527	210	248	210	253	230	55	1-3/4	1-3/4	1-3/4	1-3/4	1-3/4
20	536	572	542	570	548	225	265	230	265	248	55	1-3/4	1-3/4	1-3/4	1-3/4	1-3/4
30	550	588	568	586	563	230	275	242	280	260	55	1-3/4	1-3/4	1-3/4	1-3/4	1-3/4
40	555	595	592	590	578	235	280	248	290	263	55	2-3/8	1-3/4	1-3/4	1-3/4	1-3/4
50	576	604	620	615	595	243	282	250	293	267	55	2-3/8	1-3/4	2-3/8	1-3/4	1-3/4
60	598	616	653	627	615	250	280	257	293	270	55	2-3/4	1-3/4	2-3/8	2-3/8	1-3/4
70	635	640	672	644	648	253	275	266	292	280	55	3-1/2	2-3/4	3-1/2	2-3/4	2-3/4
80	680	676	686	675	680	258	276	268	293	288	55	4-1/4	3-1/2	4-1/4	3-1/2	3-1/2
90	-	718	-	700+	-	-	250	-	280	-	55	-	4-3/4	-	5-3/4	-

* 26.3 wt. % OMH-1 (sodium aluminum diethyl dihydride) in toluene (containing 3-4% tetrahydrofuran). Subheadings represent percent by weight OMH-1 treatment.

Table 68. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH 0.13 WEIGHT PERCENT OMH-1*

Vol. % Recovered	Still Temp., °F		Vapor Temp., °C		Vacuum Torr		ASTM Color	
	#3	#4	#3	#4	#3	#4	#3	#4
Initial	490	555	140	120	95	55		-
10	546	572	225	235	95	55	2-3/4	1-3/4
20	575	598	255	254	95	55	2-3/8	1-3/4
30	580	602	265	258	95	55	2-3/8	1-3/4
40	590	606	268	262	95	55	2-3/8	2-3/8
50	598	633	278	270	95	55	2-3/8	2-3/8
60	600	635	278	280	95	55	2-3/8	2-3/4
70	615	650	278	280	95	55	2-3/8	3-1/2
80	620	680	280	283	95	55	2-3/8	3-1/2
90	680	700+	290	275	95	55	-	6-1/2

* As 26.3 wt.% OMH-1 (sodium aluminum diethyl dihydride) in tolvene (containing 3-4% tetrahydrofuran). NORCO No. 3 and 4 distillates from run on Jan. 23, 1974.

Table 69. REDISTILLATION OF NORCO NO. 3 AND 4 BLEND (50-50 BY VOLUME) FROM JANUARY 23, 1974 OPERATION WITH OMH-1

Vol. % Recovered	Still Temp., ^{o}F		Vapor Temp., ^{o}C		Vacuum Torr		ASTM Color	
	A	B	A	B	A	B	A	B
Initial	480	500	130	120	75	55	-	-
10	548	540	225	243	75	55	1-3/4	1-3/4
20	560	558	247	255	55	55	1-3/4	1-3/4
30	566	570	255	260	55	55	1-3/4	1-3/4
40	575	588	257	270	55	55	1-3/4	1-3/4
50	584	595	257	272	55	55	1-3/4	1-3/4
60	595	613	260	278	55	55	3-3/4	1-3/4
70	596	634	260	282	55	55	2-3/4	2-3/8
80	630	690	262	288	55	55	3-1/2	3-1/2
90	670		265	-	55	55	5-3/4	-

A = 0.13 weight % OMH-1
added to blend before distillation.

B = Cuts from A reblended (ASTM Color = 2-3/4) and then
redistilled (no added OMH-1)

Table 70. REDISTILLATION OF CRANKCASE OIL DISTILLATE WITH BOROHYDRIDE PLUS OMH-1 TREATMENT -0.01 wt. % $NaBH_4$ (as 12 wt. % $NaBH_4$ in 40 wt. % NaOH water solution) added prior to distillation in each case

Vol. % Recovered	Still Temp., °F				Vapor Temp., °C				ASTM Color			
% OMH-1	-	-	-	0.13*	-	-	-	0.13*		-	-	0.13
Initial	440	430	420	540	105	90	90	60	-	-	-	-
% OMH-1	-	-	-	-	-	-	-	-	-	-	-	-
10	538	530	540	588	218	210	235	215	1-3/4	1-3/4	1-3/4	-
% OMH-1	-	-	-	-	-	-	-	-	-	-	-	-
20	562	543	562	610	232	248	247	267	1-3/4	1-3/4	1-3/4	1-3/4
% OMH-1	-	0.078	-	-	-	0.078	-	-	-	0.078	-	-
30	572	560	572	614	240	247	247	280	1-3/4	1-3/4	1-3/4	1-3/4
% OMH-1	-	-	-	-	-	-	-	-	-	-	-	-
40	578	562	585	626	235	248	250	275	1-3/4	1-3/4	1-3/4	1-3/4
% OMH-1	0.052	-	-	-	0.052	-	-	-	0.052	-	-	-
50	570	571	600	635	230	247	255	280	1-3/4	1-3/4	1-3/4	1-3/4
% OMH-1	-	-	0.078	-	-	-	0.078	-	-	-	0.078	-
60	580	588	627	644	233	246	260	282	1-3/4	2-3/8	1-3/4	1-3/4
% OMH-1	-	-	-	-	-	-	-	-	-	-	-	-
70	615	625	660	660	227	245	270	284	2-3/4	3-1/2	3-1/2	2-3/4
% OMH-1	-	-	-	-	-	-	-	-	-	-	-	-
80	685	687	718	700	240	247	260	285	3-1/2	5-3/4	4-3/4	3-1/2

* Vacuum = 85 Torr. 55 Torr in other experiments.

Table 71. REDISTILLATION OF CRANKCASE DISTILLATE HEAVY CUTS FROM PREVIOUS BOROHYDRIDE PLUS OMH-1 EXPERIMENTS WITH 0.01% SODIUM BOROHYDRIDE AND 0.13% OMH-1

FEED: 60, 70, and 80 volume percent cuts from previous experiments. ASTM color of cuts ranged from 2-1/2 to 4-1/2.

REAGENTS: 0.01 wt.% $NaBH_4$ as 12 wt.% $NaBH_4$ in 40 wt.% NaOH water solution plus 0.13 wt.% OMH-1 as 26.3 wt.% OMH-1 (sodium aluminum diethyl dihydride) in toluene (containing 3-4% tetrahydrofuran).

Vol. % Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum Torr	ASTM Color
Initial	510	90	55	-
10	565	235	55	1-3/4
20	575	245	55	1-1/8
30	580	250	55	1-1/8
40	588	252	55	1-1/8
50	600	257	55	1-1/8
60	615	260	55	1-1/8
70	638	278	55	1-1/8
80	662	283	55	3-3/4
90	700	250	55	3-1/2

Table 72. DISTILLATION OF RAW CRANKCASE OIL WITH 0.005% SODIUM BOROHYDRIDE AFTER WASHING WITH POTASSIUM HYDROXIDE

TREATMENT: Wash 200 ml. of raw crankcase oil with 10 vol. % of 5 wt. % KOH solution twice,
and centrifuge at 3000 G for 30 minutes.
Add 0.175 grams of Borex (5% $NaBH_4$ in oil).

% Recovered	Still Temp., °F	Vapor Temp., °C	Vacuum, Torr	ASTM Color
Initial Boiling Point	420	45	70	
10	546	130	70	*
20	618	255	60	-
30	622	260	60	2 3/4
40	638	270	60	2 3/8
50	655	275	60	2 3/4
60	680	277	60	3 1/2
70	712	282	60	-
80	745	273	60	-

* First cut - 10cc oil + 10cc water

Table 73. TREATMENT OF RAW CRANKCASE OIL WITH 10% KOH AND DISTILLATION WITH 0.012 % SODIUM BOROHYDRIDE

TREATMENT: Raw crankcase oil was treated twice with 5 vol. % of 10 wt. % KOH solution, centrifuged, and then distilled with 0.012 wt. % $NaBH_4$ (12 wt. % $NaBH_4$ in 40 wt. % aqueous sodium hydroxide solution).

Vol. % Recovered	Still Temp., °F		Vapor Temp., °C		Vacuum Torr	ASTM Color	
	10,000G	32,000G	10,000G	32,000G		10,000G	32,000G
Initial	420	440	90	110	60	-	-
10	515	532	165	220	60	-	-
20	566*	563	245*	255	60	2 3/8*	2 3/8
30	584	583	260	265	60	2 3/8	2 3/8
40	604	605	265	273	60	2 3/4	3 1/2
50	626	638	268	278	60	3 1/2	4 1/4
60	653	650	269	282	60	4 1/4	4 1/4
70	675	687	269	288	60	4 3/4	4 3/4
80	695	730	255	288	60	-	-

* Cut contained 40% H_2O

Table 74. DISTILLATION OF RAW CRANKCASE OIL TREATED WITH 0.24 AND 0.36% POTASSIUM BOROHYDRIDE*

% Recovered	Still Temp., °F		Vapor Temp., °C		Vacuum, Torr		ASTM Color	
	0.24	0.36	0.24	0.36	0.24	0.36	0.24	0.36
10	558	560	214	220	50	53	2 3/4	2 3/4
20	585	590	240	245	50	58	2 3/4	1 3/4
30	605	600	247	260	50	58	2 3/4	1 3/4
40	630	618	253	255	50	58	4 1/4	1 3/4
50	655	663	256	255	50	57	4 3/4	2 3/4
60	677	695	258	260	50	57	-	4 1/4
70	670	710	255	280	50	57	-	5 5/8
80	695	-	240	275	50	57	-	-

* In 12% KBH$_4$/40% KOH water solution.

Table 75. DISTILLATION OF RAW CRANKCASE OIL PRETREATED WITH 0.24% SODIUM BOROHYDRIDE#

Pretreatment: 2 wt. % soluble water solution of $NaBH_4$* at 200°F for 1 hour followed by centrifugation at 32,000 G for 30 minutes

Vol. % Recovered+	Still Temp., °F	Vapor Temp., °C	Vacuum (Torr)	ASTM Color
10	550 (558)	225 (214)	50	1-3/4 (2-3/4)
20	566 (585)	235 (240)	50	1-3/4 (2-3/4)
30	600 (603)	243 (247)	50	1-3/4 (2-3/4)
40	620 (630)	245 (253)	50	2-3/8 (4-1/4)
50	652 (655)	264 (256)	50	3-1/2 (4-3/4)
60	673 (677)	270 (258)	50	3-1/2 -
70	698 (670)	275 (255)	50	4-3/4 -
75	720 -	270 -	50	-- -
80	- (695)	- 240	50	-- -

* 12% $NaBH_4$ in 40% aqueous sodium hydroxide solution
+ First 5% recovered was water
# Numbers in parentheses are for duplicate run with no preheating and no centrifugation

Table 76. TREATMENT OF RAW CRANKCASE OIL WITH 0.36% POTASSIUM BOROHYDRIDE*-- EFFECT OF PRETREATMENT

Pretreatment Methods

A. None (KBH_4 added to still).

B. Centrifuged 15 minutes at 32,000 G. Then added KBH_4.

C. Centrifuged 15 minutes at 10,000G. Then added KBH_4.

D. Heat treatment at 200°F for 1 hour. Centrifuged at 32,000 G for 30 minutes. Decanted oil and added 0.18% KBH_4. Additional 0.18% KBH_4 added gradually during distillation.

Distillation Results

% Recovered	Still Temperature, °F				Vapor Temperature, °C				Vacuum, Torr				ASTM Color			
	A	B	C	D	A	B	C	D	A	B	C	D	A	B	C	D
10	560	510	550	575	220	160	200	215	58	57	57	55	2 3/4	1 3/4	1 3/4	1 3/4
20	590	540	563	600	245	170	220	223	58	55	55	55	1 3/4	1 3/4	1 3/4	1 1/8
30	600	550	575	615	260	194	228	235	58	55	58	55	1 3/4	1 3/4	1 3/4	--
40	618	604	610	645	255	200	235	240	57	55	58	55	1 3/4	1 3/4	1 3/4	--
50	663	648	650	668	260	243	240	240	57	55	58	55	2 3/4	1 3/4	2 3/8	--
60	695	683	705	695	280	250	260	240	57	55	58	55	4 1/4	2 3/4	4 1/4	--
70	710	695	730	718	275	254	258	235	57	55	58	55	5 5/8	3 1/2	6 1/2	--
80	-	760	-	-	-	210	-	-	-	55	-	-	--	6 3/4	--	--

---

* In 12% KBH_4/40% KOH water solution.

Table 77. REDISTILLATION OF CRANKCASE OIL BOTTOMS WITH POTASSIUM BOROHYDRIDE

Charge: 600 ml. of raw crankcase oil after centrifugation at 32,000 G for 30 minutes

Vol. Recovered	Still Temp, °F	Vapor Temp, °C
initial boiling point	250	51
5 ml.	359	67
10	410	88
20	443	106
30	476	144
40	486	144
50	518	194
60	530	224
70	end of distillation	

Bottoms recovered and 200 ml plus 4.40 grams of potassium borohydride charged to vacuum distillation

			Vacuum mm Hg	ASTM Color
initial boiling point	420	60	11.2	-
20 ml	572	180	11.0	4 7/8
40	666	210	11.0	4 1/8
60	640*	175*	9.0	4 7/8
80	660	217	5.7	4 7/8
100	690	275	5.3	4 1/8
120	725	253	5.7	4 7/8
140	750	285	5.7	5 3/4

* Restarted after problems with vacuum pump

DIESEL FUEL TESTS

The following results were obtained from a diesel engine truck test using NORCO No. 3 distillate:

Test	Ratio NORCO #3 to Reg. Diesel	Total Gals. Used	Miles Per Gallon
1	50/50	137	4.0
2	75/25	103	6.0
3	100/0	134	6.2

No foreign matter or varnish was detected in the fuel filter system. However, traces of black smoke were emitted from the exhaust pipe and a very objectionable odor was noted.

In a test with a second trucking company, 3 diesel trucks used NORCO #3 as a fuel for distances up to 500 miles. Engine inspections after the tests showed some tar deposition. Black smoke was noted intermittently in these tests, especially on very short runs.

It was concluded that light distillate recovered from crankcase waste oil can be used as a diesel fuel, but that further treatment of the distillate is necessary. A lengthy laboratory and field testing program would be necessary to develop this market. A diesel test report is provided in Appendix F.

It should be noted that there is apparently no tax incentive for converting waste lubricating oil to diesel fuel, based on the following quotation from the Internal Revenue Regulations, Regulation Section 48.4091-2(b)(2):

> "For purposes of the tax imposed under Sec. 4091, the term "manufacturer" does not include: (i) any person who merely blends or mixes two or more taxable oils, (ii) any person who merely cleans, renovates, or refines used or waste lubricating oil, or (iii) any person who merely blends or mixes one or more taxable oils with used or waste lubricating oil which has been cleaned, renovated, or refined,...."

SECTION VI

DESIGN AND ECONOMIC STUDIES

The design and economic studies reported here were undertaken to help guide research and planning aimed at the development of a process or processes for conversion of waste oils to useful products, while eliminating or minimizing waste products which contribute to environmental pollution. In NORCO's previous work, it had been shown that vacuum distillation could be used to convert crankcase waste oil to naphtha, useful as a fuel; to distillates, potentially useful as lube stock, but suffering from stability, color, and odor problems; and a bottoms fraction with questionable application.[1]

In the present work reported in Sections IV and V, it was shown that the distillates could be upgraded by catalytic hydrogen treatment and that the bottoms could be used in secondary lead smelting, thus technically meeting the objective of producing useful products without simultaneously producing wastes. At the same time, it was shown that vacuum distillation could be used to upgrade a broad spectrum of waste oils, especially those contaminated with water. The following information verifies that these technical innovations are economically feasible.

PROCESS SCREENING STUDIES

The analysis of process options open to NORCO was complicated by the availability to NORCO of used vacuum distillation and catalytic hydrotreating equipment from Exxon's Bayonne facilities. This equipment, which had been used in wax processing service, has a nominal capacity of about 3500 barrels per stream day. Therefore, the screening studies, provided in Appendix J, consider both relocating the Exxon equipment and building a grass roots plant. The results can be summarized as shown on Table 78.

It was concluded from this study that:

1. There is a strong economic incentive to develop processes for refining crankcase oil to produce saleable lube oils, when there is a spread on the order of 13¢/gal. between crankcase oil and lube oil.

Table 78. SCREENING STUDY ECONOMICS

Pre-Treat.	----------None----------------			Solvent
Post-Treat.	--------Hydrogen-----------------------			
Equip.	Exxon	Exxon	New	Exxon
Feed, 10^6 gals/yr.	9	29	29	34.8
Invest. $\$10^6$	0.49	1.12	3.05	1.77
Profit $/yr.	0.129	1.57	1.49	2.19
Return, %/yr. before tax	26	140	49	124

Notes:
1. Crankcase waste oil at 3¢/gal.
2. Lube product at 16¢/gal.
3. Study date - January 1973

2. Investments and operating costs projected for vacuum distillation and hydrotreating can easily be justified if they can produce 60% or greater yield of lube oil.

3. Additional investment for solvent pretreatment can be justified, if yield of lube oil is improved.

4. Based on knowhow available from the petroleum refining industry, the prognosis for technical and economic success of a vacuum distillation/catalytic hydrotreating process is highly favorable.

Later studies (June 1973) showed that either relocation of the Exxon equipment or a grass roots (new) plant processing about 29 million gallons per year of crankcase waste oil and/or other waste oils is a potentially profitable venture (Tables 79-82).

A subsequent study showed that the vacuum distillation/catalytic hydrogen treatment process compared favorably with other re-refining processes in a 5 million gallon per year plant producing lube blending stocks.[8,9] Of the processes available, only the hydrotreating approach produces no waste products (Table 83).

The principal economic problem encountered today in any re-refining operation is competition for feedstock with indiscriminate use as a fuel or for road oiling, both of which contribute to environmental pollution. One solution to this problem is governmental regulations to control the burning of waste oils which have high metals contents (including all automotive crankcase waste oils, many of which contain more than 1% lead). Another important aid is the development of superior re-refining technology (e.g., hydrotreating) in large plants, which is sufficiently attractive at current (1975) lube stock prices (40-60 cents per gallon) to allow at least 10-15 cents per gallon payment to collectors for the waste oil (or which provides an incentive for collection by the re-refiner).

Table 79. CASE DESCRIPTIONS

CASE	PURCHASE EXXON MONOPHINER + VACUUM FRACTIONATOR 1	2	3	GRASS ROOTS PLANTS 4	5	6	7	8
Feed, MMGPY								
Crankcase Oil	9.0	14.5	29.0	9.0	14.5	29.0	14.5	20.5
Fuel Oil - 10% BS&W	20.0	14.5	----	20.0	14.5	----	14.5	14.5
	29.0	29.0	29.0	29.0	29.0	29.0	29.0	35.0
Vac. Fract.								
B/SD	3314	3314	3314	3314	3314	3314	4000	4000
Hrs/Yr.	5000	5000	5000	5000	5000	5000	4143	5000
Hydrotreat.								
B/SD	2117	2117	2117	657	1059	2117	1497	1497
Hrs/Yr.	1552	2500	5000	5000	5000	5000	3537	5000
Incremental Investment, $M								
Exxon Equip.	----	889	----	--	--	--	--	--
New Vac. Dist.	----	----	----	850	850	850	940	940
New Hydrotreating	----	----	----	530	740	1200	930	930
New H_2 Plant	----	----	----	240	320	470	380	380
Other	----	611	----	1480	1480	1480	1700	1700
	----	1500	----	3100	3390	4000	3950	3950

Table 80. FEED AND PRODUCTS

CASE	1	2	3	4	5	6	7	8
Feed, MMGPY								
Crankcase Oil	9.0	14.5	29.0	9.0	14.5	29.0	14.5	20.5
Fuel Oil	20.0	14.5	----	20.0	14.5	----	14.5	14.5
	29.0	29.0	29.0	29.0	29.0	29.0	29.0	35.0
Net Products, MMGPY								
Lube Oil	5.75	9.26	18.52	5.75	9.26	18.52	9.26	13.09
Light Cuts	-----	-----	0.82	-----	-----	0.82	-----	-----
Pb Sludge	1.96	3.17	6.34	1.96	3.17	6.34	3.17	4.48
Fuel Oil	17.60	12.90	-----	17.60	12.90	-----	12.90	12.92
	25.31	25.33	25.68	25.31	25.33	25.68	25.33	30.49

Table 81. PROFITS

CASE	1	2	3	4	5	6	7	8
	---Relocate Exxon Equip.---			---Grass Roots---				
% Hydrotreat. Cap. Used.	31.0	50.0	100	100	100	100	70.7	100
Feed, MMGPY								
Crankcase Oil	9.0	14.5	29.0	9.0	14.5	29.0	14.5	20.5
Fuel Oil	20.0	14.5	----	20.0	14.5	----	14.5	14.5
10% BS&W	29.0	29.0	29.0	29.0	29.0	29.0	29.0	35.0
Feed Costs, $M/Yr.								
Crankcase Oil @ 5¢/G*	450	725	1450	450	725	1450	725	1025
Fuel Oil @ 1¢/G*	200	145	----	200	145	----	145	145
	650	870	1450	650	870	1450	870	1170
Oper. Costs,$M/Yr.	851	925	1090	768	820	920	875	902
Total Costs,$M/Yr.	1501	1795	2540	1418	1690	2370	1745	2072
Revenues, $M/Yr.								
Lube Oil @ 20¢/G	1150	1852	3704	1150	1852	3704	1852	2618
Light Cuts @ 10¢/G.	----	----	82	----	----	82	----	----
Pb Sludge @ 2¢/G	39	63	127	39	63	127	63	90
Fuel Oil @ 10¢/G	1760	1290	----	1760	1290	----	1290	1292
	2949	3205	3913	2949	3205	3913	3205	4000
Profit B.T.,$M/Yr.	1448	1410	1373	1531	1515	1543	1460	1928
Profit After 50% Tax, $M/Yr.	724	705	686.5	765.5	757.5	771.5	730	964
Return A.T., %/Yr.	48.3	47.0	45.8	24.7	22.3	19.3	18.5	24.4

---

*Includes transportation costs, if any.

Table 82. COSTS

CASE	1	2	3	4	5	6	7	8
	--Relocate	Exxon	Equipment--	----------	Grass	Roots	----------	----------
Feed, MMGPY								
Crankcase Oil	9.0	14.5	29.0	9.0	14.5	29.0	14.5	20.5
Fuel Oil	20.0	14.5	----	20.0	14.5	----	14.5	14.5
	29.0	29.0	29.0	29.0	29.0	29.0	29.0	35.0
% Hydrotreat. Cap. Used	31.0	50.0	100	100	100	100	70.7	100
Dir. Op. Costs, $M/Yr.								
Op. Labor	110.0	110.0	100.0	110.0	110.0	100.0	110.0	110.0
Labor O.H.	16.5	16.5	15.0	16.5	16.5	15.0	16.5	16.5
Ins. + Taxes	60.5	60.5	60.5	60.5	60.5	60.5	60.5	60.5
Catalyst	14.7	23.7	47.3	14.7	23.7	47.3	23.7	33.4
Antifoulant	2.3	3.7	7.3	2.3	3.7	7.3	3.7	5.2
Hydrogen	80.5	129.7	259.3	----	----	----	----	----
Maint.	78.2	78.2	78.2	93.0	101.7	120.0	118.5	118.5
Deprec.	233.5	233.5	233.5	206.7	226.0	266.7	263.3	263.3
Power	23.1	35.0	53.6	20.0	25.0	31.0	25.0	32.0
City Water	2.0	4.0	5.2	3.0	5.0	7.0	5.0	8.0
Nat. Gas	----	----	----	10.9	17.5	35.1	17.5	24.8
	621.3	694.8	859.9	537.6	589.6	689.9	643.7	672.2
¢/Gal. Feed	2.14	2.40	2.97	1.85	2.03	2.38	2.22	1.92
Indir. Op. Costs, $M/Yr.								
Salaries	143.0							
Salary O.H.	22.0							
R & D	25.0							
Lab. & Office Exp.	15.0							
Consultants & Other	25.0							
	230.0	230.0	230.0	230.0	230.0	230.0	230.0	230.0
¢/Gal. Feed	0.79	0.79	0.79	0.79	0.79	0.79	0.79	0.66

Table 83. SUMMARY OF CRANKCASE WASTE OIL PROCESSES*

Process	Primary Product	Primary Wastes & Byproducts	Grass Roots Econ.- 5 Million Gal/Yr. Investment	Grass Roots Econ.- 5 Million Gal/Yr. Op. Cost*	Comments
Acid/Clay	Lube blending stock	Acid sludge, spent clay	$1,153,000	21.9¢/Gal. Lube	Widely used in U. S.
Extraction/ Acid/Clay	Lube blending stock	Acid sludge, spent clay; high ash fuel byproduct	$1,363,000	18.4¢/Gal. Lube	One operating plant in Italy.
Distillation/ Clay	Lube blending stock	Spent clay; high ash fuel byproduct	$1,173,000	17.3¢/Gal. Lube	At least two plants in U.S.
Distillation/ H_2 Treating	Lube blending stock	High ash fuel byproduct	$1,342,000	19.0¢/Gal. Lube	Under development.
Distillation	Fuel oil (diesel fraction could possibly be recovered)	High ash fuel byproduct	$ 930,000	14.6¢/Gal. Fuel oil	Can make high quality fuel, but economics questionable.
Controlled Combustion	Steam	Ash concentrate	$ 492,000	80¢/1000 Lbs. Steam	Speculative-fine particle recovery difficult.

* Includes 3¢/gal. feed cost and 10%/yr. depreciation, but excludes return on investment. See Tables 9,10, and 11 for details.

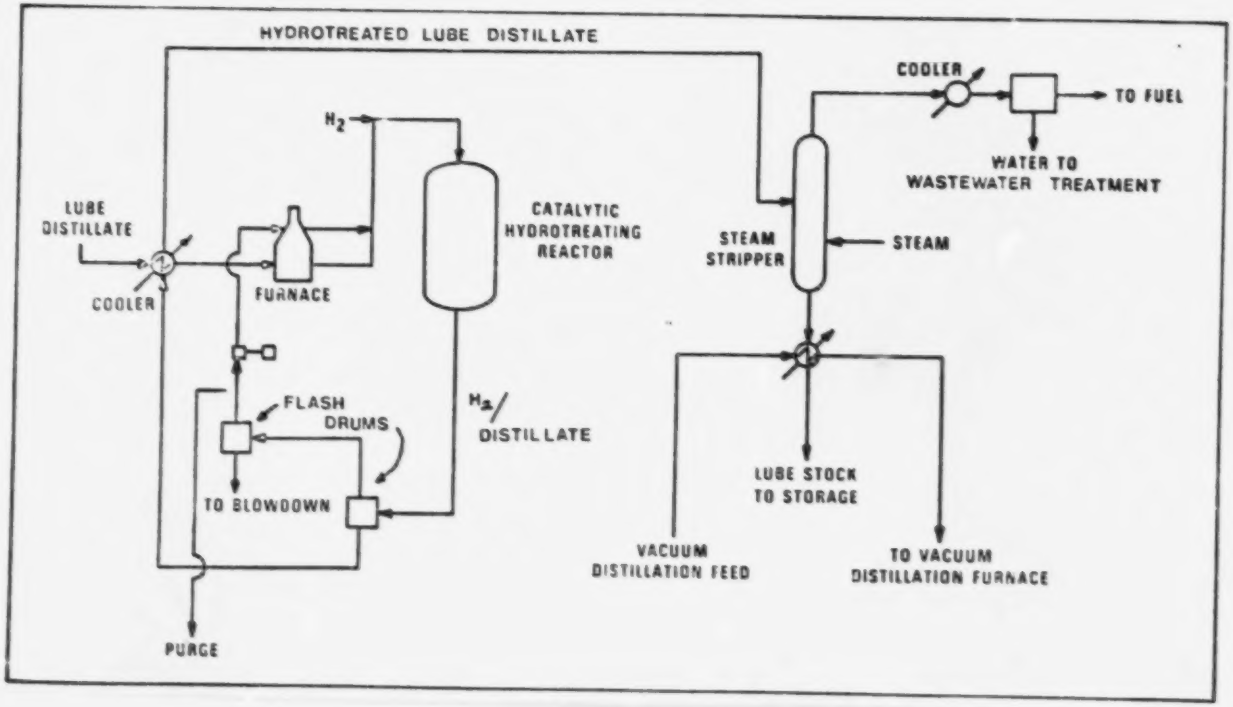
VACUUM DISTILLATION/HYDROTREATING PROCESS

The basic scheme for the distillation section of the process was provided in Figure 1; the hydrotreating section is shown in Figure 9. The raw crankcase oil is first run through a flash column where water and some of the gasoline contamination is taken overhead. The flash bottoms are then processed through a vacuum distillation column separating the following components: gasoline contamination overhead; a vacuum distillation bottoms product, containing high boiling hydrocarbons and non-volatiles including metallic components; and one or two distillate fractions. The distillate is dark in color and otherwise unsuitable for use as a high quality lubricant.

The distillate is then catalytically hydrogen treated to meet specifications as a lubricating oil blending stock. Work reported by Esso Research and Engineering Co. shows that hydrogen treated distillate can match typical properties of 150 vis neutral lube base stock.[3] Other approaches to hydrogen treatment have been reported in patent literature.[10]

The gasoline fraction can be used internally as a fuel, and the balance sold. The vacuum distillation bottoms, which may contain more than 10% lead, can be used as fuel in secondary lead smelting furnaces, as reported in Section V.

The principal problems in catalytic hydrogen treating left unanswered by previous work are the questions of catalyst life and hydrogen consumption. In Esso's work, about 100 hours of continuous hydrogen treating of the used motor oil distillate was logged with no noticeable catalyst deactivation. In the experiments reported in Appendix E, no problems were noted after 67 hours of operation. At a lead content of 2 ppm in the distillate and 1.0 V/V/hr. space velocity, the catalyst bed would contain about 1.5% lead after one year of operation, if breakthrough did not occur. Actually breakthrough would be expected, and, in any case, this lead level would not be expected to seriously affect the catalyst.


HYDROTREATED LUBE DISTILLATE
LUBE DISTILLATE
COOLER
FURNACE
H_2
CATALYTIC HYDROTREATING REACTOR
H_2/ DISTILLATE
FLASH DRUMS
TO BLOWDOWN
PURGE
STEAM STRIPPER
STEAM
COOLER
TO FUEL
WATER TO WASTEWATER TREATMENT
LUBE STOCK TO STORAGE
VACUUM DISTILLATION FEED
TO VACUUM DISTILLATION FURNACE

VACUUM DISTILLATION/HYDROTREATING PROCESS
HYDROTREATING SECTION

FIGURE 9

Hydrogen consumption is a serious matter, since hydrogen costs for the relatively small quantities required are on the order of \$4 per 1000 standard cubic feet. Accurate measurement of hydrogen consumption in small pilot units is very difficult and expensive. Therefore, two different approaches have been used to estimate the hydrogen which will be consumed. (See Appendix E for details).

In one approach a static hydrogenation bomb test for distillate was compared to a bomb test for Nujol at the required temperature and pressure. This data, after being subjected to corrections involving catalyst reduction, ammonia, hydrogen sulfide, and water formation, and sorption phenomena, gave a maximum hydrogen consumption of 70 to 160 SCF/B.

In an alternative approach, hydrogen consumption was calculated from changes in the characteristics of distillates during catalytic hydrogen treating. The characteristics examined include sulfur, nitrogen, and oxygen contents, hydrogen content as predicted from specific gravity changes, and Iodine Number (unsaturation). This approach led to a predicted hydrogen consumption of 153 SCF/B, in reasonable agreement with the values cited above. Hydrogen makeup cost at the 150 SCF/B level is about 1¢/gal. of raw waste oil (\$4 per 1000 standard cubic feet for hydrogen).

WASTEWATER TREATMENT

The existing wastewater treatment system consists of a primary oil separator drum, and two oil/water gravity separator tanks arranged in series for treatment of all process and cooling waters prior to discharge to the Kill Van Kull. Oil is recovered by the use of vacuum hoses, and solids are removed by periodic cleaning of the oil/water separators. The planned addition of a new process unit will increase plant capacity by approximately 3500 bbls. per day from the current capacity of 1000 bbls. per day and will necessitate upgrading of the present waste water treatment system.

The improved treatment facility will include holding areas for process water and rainwater runoff, and the addition of a dissolved air flotation unit downstream of the existing separators. The quality of effluent is expected to be significantly improved by (1) the additions to the physical facilities of the treatment plant referred to above and (2) reduction in the quantity of wastewater requiring treatment by a factor of 9 (43.8 gpm vs. 420 gpm). Figure 10 is a flow plan of the upgraded treatment facility.

Holding Areas

The holding ponds and contaminated water storage tanks provide a reliable method for maintaining a constant flow rate to the treatment plant, and permit the collection and treatment of storm water. In addition, some oil and suspended solids removal can be achieved in the holding ponds and tanks before the impounded water is fed to the treatment plant.

No. 1 Pond has a holding capacity of 27,000 gallons. The No. 1 Pond is the main collection basin used to feed the treatment plant, and is located for easy access to all processing units, truck loading facilities and tankage. Contaminated water will flow by gravity from the storage tanks to the pond. A pump is provided to pump water from the pond to the contaminated water storage tanks during heavy precipitation.

No. 2 Pond has a holding capacity of 6,000 gallons. The No. 1 Pond is a secondary collection basin used for control of storm water collected at the South end of the property, and for storm water in the No. 1 Tank Area. Water from the No. 2 Pond is pumped to the No. 1 Pond or to the contaminated water storage tanks prior to being fed to the treatment plant.

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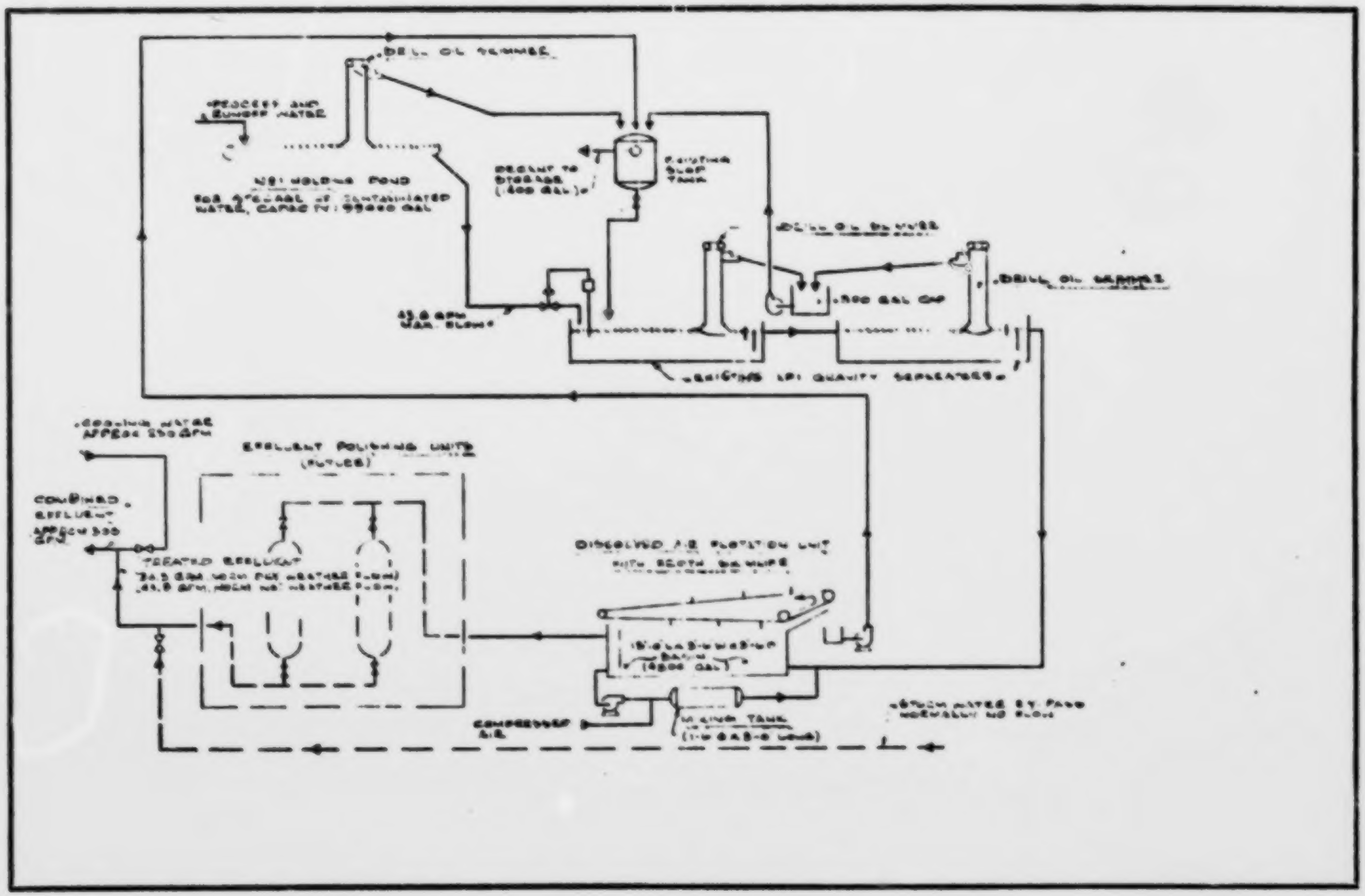
DRILL OIL SKIMMER
PROCESS AND RUNOFF WATER
NO 1 HOLDING POND
FOR STORAGE OF CONTAMINATED WATER, CAPACITY: 99000 GAL
DECANT TO STORAGE (1400 GAL)
EXISTING SLOP TANK
43.8 GPM MAX. FLOW
DRILL OIL SKIMMER
500 GAL CAP
DRILL OIL SKIMMER
EXISTING API GRAVITY SEPARATORS
COOLING WATER APPROX 250 GPM
EFFLUENT POLISHING UNITS (FUTURE)
COMBINED EFFLUENT APPROX 300 GPM
TREATED EFFLUENT
34.3 GPM NORM FAIR WEATHER FLOW
43.8 GPM MAX WET WEATHER FLOW
DISSOLVED AIR FLOTATION UNIT WITH FROTH SKIMMERS
FLOTATION BASIN (4500 GAL)
COMPRESSED AIR
MIXING TANK
STORM WATER BY PASS NORMALLY NO FLOW

WASTEWATER TREATMENT FLOW DIAGRAM

FIGURE 10

Contaminated Water Storage Tanks (2) are provided for storage of process and runoff waters. Each tank is 20 ft. in diameter and 20 ft. high with a capacity of 47,000 gal.

Oil/Water Separators

The two oil/water separators are arranged in series for treatment of wastewater and storm water from the No. 1 Pond. The two separators are of identical design, each having a water holding capacity of approximately 7300 gallons. They consist of a hemispherical section of a cylindrical tank 29 feet in length and 10'3" in diameter. The liquid at the center of the tank is approximately 52 inches deep. Each of the two tanks are gravity fed and are baffled at the downstream (South) end for retention of oil for removal by skimming.

Air Flotation Unit

After leaving the two separators the wastewater receives additional treatment in an air flotation unit which consists of a central treatment chamber equipped with a mechanical scraper for removing oily froth from the surface, an entrance section, a discharge section and auxiliary equipment for the introduction of compressed air with the feed water. The air flotation unit is approximately 13 feet long x 3.5 feet wide x 3.5 feet liquid depth. The auxiliary equipment consists of a recirculation pump and a tank for the introduction of compressed air. As very small air bubbles are released in the central chamber, they carry oil droplets and solid particles to the surface. These are removed by the mechanical scraper and transferred to a froth drum where addition of heat or chemicals eliminates air before transferring to the slop tank. Coagulant chemicals and coagulant aids are added as necessary to the feed in order to achieve maximum efficiency in removal of oil and solid particles. It is expected that the hydrocarbons reaching the air flotation unit will have a low vapor pressure and will not cause significant hydrocarbon emissions to the air. If this should prove to be a problem,a controlled vent system might be required.

Continuous Oil Skimmers

Skimmers utilizing continuous floating collector tubes (Brill oil skimmers--Model T-6) are installed at the effluent weirs of the No. 1 Holding Pond, and in each of the two separators. Continuous skimming will prevent the build-up of significant quantities of oil on the water surface and will thus prevent reentrainment and carry-over to downstream treatment units. Recovered oil from the skimmers is directed to the slop tank for reprocessing.

Run-Off Water

The entire system is designed to prevent inadvertent draining of run-off waters to the Kill. This is accomplished by the use of curbing and drainage ditches around the NORCO site. Run-off waters due to precipitation or washing operations are collected in (1) one of two holding ponds, (2) process area sumps, (3) behind tank area curbing, and (4) the loading area sump. The ultimate destination of all these waters, either by drainage or pumping, is the No. 1 Holding Pond. The water from the No. 1 Holding Pond is pumped at a controlled rate through the wastewater treating system described in the previous section, and finally the clean effluent is discharged to the Kill.

The system designed handles all rainfalls up to approximately 1 1/3 inches over a one hour period. In the event of an extremely heavy rainfall in excess of this figure, means are provided for diverting clean water run-off directly to the Kill, bypassing the wastewater treatment system. This operation is done by closing gates which serve as inlets to the two holding ponds, preventing additional water from overflowing into or out of the full ponds.

Flow Measurement and Sampling

A flow measurement and sampling box will be provided at the effluent outflow to monitor discharges.

Additional Information

The design basis, sizing of major equipment, and projected water effluent quality are provided in Tables 84-86.

Table 84. NORCO WASTE OIL RECYCLING OPERATIONS UPGRADING OF WASTEWATER TREATING FACILITIES

DESIGN BASIS

1. The steam jets and barometric condenser for the existing 1000 B/D vacuum flash tower will be replaced with an indirect condenser and mechanical vacuum pump.

2. The steam jet condensers and barometric condenser for the existing 1000 B/D vacuum distillation tower will be replaced with an indirect condenser and mechanical vacuum pump.

3. The steam jets and barometric condenser for the relocated 3500 B/D vacuum distillation tower will be replaced with an indirect conderser and mechanical vacuum pump.

4. The steam jets and barometric condenser for the relocated vacuum stripper will be replaced with an indirect condenser and mechanical vacuum pump.

5. All cooling services in the plant will be by indirect heat exchange with air or cooling water. The cooling water will be pumped from the Kill Van Kull, and used once-thru. An oil detector will be used at the discharge to detect leaks. Leak detection will result in diversion of the water to retention ponds or shutdown of the leaking equipment. A higher pressure on cooling water than on oil will cause leakage of water into oil rather than oil into water.

6. All process water (water contained in and recovered from waste Oils) will be directed to the oil/water separation system.

7. All normal runoff water will be directed to the oil/water separation system, up to 1 1/3 inches rainfall in a one hour period. Excess runoff in very heavy storms will bypass the separation system.

8. Basic calculations -

   Runoff

   3.5 acres* x 43,560 ft^2/acre x 12 = 12,750 ft^3/inch rainfall

---

*Note total site area is approximately 3.5 acres. About 25% of total area covered by diked or curbed process and tank areas. Therefore, one inch of precipitation over 3.5 acres is equivalent to $\frac{1}{0.75}$ = 1 1/3 inches over free area.

Table 84. (Continued).

Waste Water Holding Areas

Holding Pond No. 1 - 3,600 ft^3 (27,000 gal.)

Holding Pond No. 2 - 800 ft^3 (6,000 gal.)

4,400 ft^3 (33,000 gal.)

Contaminated Water Storage (2 Tanks) - 94,000 gal. (12,600 ft^3)

Run-off Treatment Rate (Based on 1 inch over 7 days)

12,750 ft^3 over 7 day period

$$\frac{12,750\ ft^3 \times 7.48\ gal/ft^3}{7\ day \times 1440\ min./day} = 9.5\ GPM$$

Process Water - Maximum

3500 B/D Crankcase Oil @ 5% water = 5.1 GPM

2000 B/D Other Waste Oils @ 50% water = 29.2 GPM

34.3

Wastewater Treatment Plant - Design Basis

Run-off Water - 9.5 GPM
Process Water - 34.3 GPM

TOTAL 43.8 GPM

9. The wastewater system will be designed and installed in two phases. Phase I equipment is described in this plan to meet target water effluent quality shown in the following document. The Phase II design will depend upon the performance of the Phase I system and applicable advances in wastewater treatment equipment.

Table 85. SIZING OF MAJOR TREATMENT PLANT UNITS
National Oil Recovery Corporation
Bayonne, New Jersey 07002

No. 1 Holding Pond (27,000 gal.)

This pond was designed to contain the process water resulting from normal operations and, with the contaminated water storage tanks, to have sufficient capacity for containing the first 1 inch of rainfall collected from the 3.5 acre site. Since approximately 25% of the site contains separate enclosures, capacity is about 1.33 inches of rainfall. The capacity provided will handle normal daily rainfall, with bypassing required only occasionally (usually not more than once in a given month).

No. 2 Holding Pond (6,000 gal.)

This pond was designed to collect storm water run-off from the South portion (approximately 3/4 acre) of the property which is too low in elevation to drain into the No. 1 Holding Pond. Pond 2 therefore is used for pumping to Pond 1. In addition, storm water from the No. 1 Tank Area can be manually discharged to Pond 2 for pumping to the No. 1 Holding Pond. A 500 GPM pump will be provided.

Oil/Water Separators

At the design treatment rate of 43.8 GPM, and the liquid holding capacity of 7300 gallons, each separator has a detention time of 166 minutes (2.75 hours). The detention time for both separators is therefore 5.5 hours. Accepted design practice for gravity separators utilizes detention time of approximately 4 hours to achieve maximum oil-water separation efficiency.

Air Flotation Unit

Based on the design treatment rate of 43.8 GPM and the liquid holding capacity of 1200 gallons, the detention time in the flotation unit is approximately 27 minutes. Recommended detention times ranging from 4 minutes up to the time used were obtained from various manufacturers of flotation equipment. A second parameter used to size flotation equipment is the overflow rate which is defined as the throughput

Table 85. (Continued)

rate (GPM) divided by the liquid surface area of the central flotation chamber (sq.ft.). The overflow rate of the unit described is 43.8/25 or 1.75 which is more conservative than the accepted value of 2 used for design of most refinery units in service today. Proprietary information based on pilot plant studies in a refinery show that a flotation unit designed using these criteria and using chemical additions will remove 80 to 90 percent of the suspended solids present in the feed and will reduce oil content to the range of 10-40 mg/l. The oil content of the combined outflow (with cooling water) is expected to be less than 6 ppm.

Continuous Oil Skimmers

The capacity of the Brill Model T-6 oil skimmer is rated by the manufacturer approximately as follows:

heavy residual oil	2500 gallons/day
lubricating oil (SAE 20)	500 gallons/day
fuel oil (No. 2)	250 gallons/day

At the design wastewater flow of 43.8 GPM, and assuming as a liberal estimate that the water entering the pond contains 0.5% oil, the daily removal requirement approximates 400 gallons of oil. The three skimmers provided can easily remove that amount of oil.

Table 86. **PROJECTED WATER EFFLUENT QUALITY**

	Present Limitation[1]	Required Effluent Discharge[2]	Target Effluent Discharge[3]		
	Avg. (Max.)	Avg. (Max.)	Avg. (Max.)	mg/l[4]	mg/l[5]
pH	6-9	6-9	6-9		
TOC	1200 lbs/day	72 lbs/day (140)	68 lbs/day	130	19
O&G	3300 lbs/day	36 lbs/day (49)	21 lbs/day	40	6
TSS	54 lbs/day	54 lbs/day (140)	32 lbs/day	60	9
Temp.	100°F (110)	100°F (110)	100°F (110)		
Zn - Total	(1 mg/l)	(1 mg/l)	--		
Cr - Total	(1 mg/l)	(1 mg/l)	--		

(1) Initial Effluent Limitation, Aug. 31, 1974 - Aug. 31, 1976, NPDES Permit No. NJ0003565
(2) Required Effluent Discharge, Aug. 31, 1976 - Aug. 31, 1979
(3) For Phase I plant modifications, about May 1976
(4) Based on discharge from air flotation unit - 43.8 GPM
(5) Based on total discharge to Kill (including cooling water) - 300 GPM

SECTION VII

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APPENDIX A

SUMMARY LOG OF OPERABILITY - PROBLEMS AND SOLUTIONS

2/15 - 3/15/74	The oil processed contained a high percentage of water (30.1% average), considerable very abrasive gritty material, and troublesome fibrous trash. The high water content required excessive recirculation of partially dewatered oil back to the feed tanks to produce dry oil. The fibrous trash passed through relatively fine strainers fitted with cylindrical basket sheet metal elements with 1/8 inch diameter holes. The fibrous trash plugged inlets of channels in the closed impellers of the two stage centrifugal pump. The single stage open impeller centrifuggal pump remained clear. Erosion on the impellers was not severe. The high grit content of the oil cut the sliding vanes in a rotary positive displacement in about 3 hours. Elements of rotary screw and rotary gear positive displacement pumps were out in about five hours. Heater erosion which occurs due to grit can be reduced by reducing velocity, e.g. operating at less vacuum or even under pressure.
1/15 - 2/15/74	A procedure was worked out whereby waste oil feed is first pumped from a warmed up feed tank through cold suction lines with a rotary positive displacement pump through a centrifugal pump until the suction lines and the centrifugal pump are adequately warmed up and filled with relatively low viscosity feed going into the fractionator heater. After about 10-15 minutes, the rotary pump is shut down and the centrifugal pump takes over. This procedure eases startup problems and reduces the erosion and abrasion wear on the rotary pump which has been experienced due to the grit content of the feed (the centrifugal pump with a priming vacuum pump is an alternative).

12/15/73-1/15/74	With the intake of a greater variety of waste oils from more sources, including vacuum tank trucks used for cleaning out tank bottoms, cleaning up spills, etc., a great increase in trash, debris, grit, metal particles, sand, plastic, and other foreign material have been noted. Swing strainers with strainer baskets are presently being employed. Slant bottom receiving tanks and strainers operating with continuous discharge should be considered for future operations.
11/15 - 12/15/73	The dismantled 2 inch pipe coil coolers for fractionator bottoms, light cut, light reflux, heavy cut, and heavy reflux were inspected after steam and air blowing to remove oil and tar. All coolers were found to be relatively clean inside, with very little tar or signs of fouling. The outside of pipe coils close to the bottom of the cooling tub was rather heavily coated with deposit from the salt cooling water. All coils were corroded rather evenly on the outside, with the 180° bends corroded more severely than the straight pipe. Corrosion varied from about 3/32 inch to 1/16 inch, with 1/8 inch on outer curve of bends and some areas corroded even deeper, accounting for leaks. Sharp localized pitting did not occur. Bronze veins tried on a rotary sliding vane pump, instead of the recommended plastic vanes, lasted somewhat longer but the wear on the surfaces of the vane containing slots in the eccentrically positioned rotor was much more rapid. The rotor had to be replaced. The wear and cost is excessive when the sliding vane rotary pump is used for pumping gritty oil.
10/15 - 11/15/74	Tube ends in the fractionator heater were sweating (leaking very slightly at ends where they are rolled into headers). Inspection showed end of 6 tubes required re-rolling. Tubes tend to leak at rolls when running waste fuel oil as heat input, pressure, erosion, and fouling rates are all increased over corresponding rates when processing waste crankcase oil.

10/15 - 11/15/74 (continued)	One leak developed when the flash heater 3 inch pipe coil was tested. In the process of patching the leak, heavy fouling of the coil and thinning of metal by outside oxidation from high metal temperature experienced during runs was noted. Two more leaks developed on retesting. The coil required replacement.
9/15 - 10/15/74	A revamp of the piping between the charge pumps and the fractionator heater, including installation of a larger orifice, reduced pressure drop and improved control of charge rate and heater outlet temperature.
8/15 - 9/15/73	Tubes in the fractionator heater were cleaned from one end with air driven tube cleaners, reducing cleaning time. Some of the tubes are exposed to excessive radiation because of poor furnace design, resulting in excessive metal temperature and blown tubes. Frequent cleaning aids in minimizing this problem. High firing rates used to increase throughput of very wet oils aggravates these problems.
7/15 - 8/15/73	40°API naphtha added to wet waste fuel oil to aid in settling and separating water in the storage tanks before vacuum distillation.
6/15 - 7/15/73	The casing of a rotary positive displacement sliding vane feed pump and the vane slots in the round rotor became excessively worn after less than three months operation on an abrasive waste fuel oil stock. The pump was junked. A procedure was worked out to clean the fractionator heater tubes from one end only, leaving the foreign material in the tubes to be removed by pumping cold oil through at high flow rates followed by steam. The foreign material passes through the tubes and the transfer line to the bottom of the fractionator where considerable volume is available for solids accumulation. The bottom of the fractionator

	is cleaned out periodically to avoid carryover of solids to the bottoms pump. The procedure reduces cleaning time and cost.
5/15 - 6/15/73	An anti-foulant from Nalco Chemical Co. was injected into the crankcase oil charge and into the light and heavy lube oil cuts at the rate of 50 ppm, with results very similar to those obtained when an anti-foulant from Exxor was injected in 1969. Fouling in the heater tubes and in the light and heavy lube oil cooling coils appeared to be reduced. The color of these two products was slightly darkened from about L 7.0 to L 7.5 or L 8.0 on the ASTM color scale. Tarry material which settles to the bottom of sample bottles did not solidify and tenaciously adhere as occurs without anti-foulant injecting. Odor seemed somewhat reduced. The Nalco anti-foulant is considerably less viscous than that from Exxon and required a different injector. Both bronze and composition vanes have been tried in rotary displacement sliding vane pumps. With bronze vanes, the wear on the vanes is slower than with composition vanes, but the wear on the slots in the cast iron rotor is greater. Overall economics favor composition vanes, but temperature must be limited to 120°F.
4/15 - 5/15/73	Some high water (50-55%) fuel oil was received. Waste oil containing over about 35-40% water cannot be economically and satisfactorily processed in a single pass through the existing equipment because of pressure drop and excessive erosion at high velocities in heater tubes and headers. Therefore, about 20 hours during the month was spent recycling the high water content fuel through the fractionator to bring the oil to dryness. Some recycling of feedstock is required to start up and shut down. This is being done in a manner to get the most evaporation of water possible, before going on stream to produce

dry products. A substantial portion of the water content of the oils is evaporated at relatively low temperature, but, since the solubility of water in oil increases with temperature, to achieve adequate drying of finished oil, the bottom of the fractionator must be maintained at a temperature above the boiling point of water, usually 40-50°F, at the absolute pressure existing at the liquid level.

3/15 - 4/15/73 Waste fuel oils from Butterworthing (washing of barge and ship tanks) processed from Nov. 1971 to Dec. 1972 did not contain much abrasive solids. Positive displacement rotary gear pumps and rotary sliding vane pumps were reasonably satisfactory for pumping this material. Since accepting tank bottoms and miscellaneous waste oils from various sources, the content of abrasive solids has increased and the wear on the pumps has been excessive. The vanes in a feed pump had to be replaced after 50 1/2 hours operation. The steam simplex pump used for pumping dried processed fuel oil from the bottom of the vacuum fractionator runs at a piston speed of 30-50 feet per minute. Wear on the piston, piston rings, and cylinder appears to be reasonably low, but the initial and operating costs for such a pump is high.

The pressure drop from feed tanks to pump suctions is often so high as to cause very low suction pressures resulting in erratic pump operation. Tank heating to lower viscosity is expensive, so that other solutions such as the use of positive displacement pumps and locating pumps immediately adjacent to tanks are being tried.

Six tubes in the fractionator heater were replaced because of local overheating and actual failure of four tubes.

	The stainless steel thermowell in the 4 inch section of the transfer line eroded through and leaked. It was replaced with a hard-faced hardened steel thermowell. The 4 inch x 4 inch x 6 inch steel tee installed to replace a 4 inch elbow in the fractionator heater transfer line was inspected after 190 hours of service. Little erosion was noted, certainly considerably less than previously noted.
2/15 - 3/15/73	Severe erosion of the fractionator heater tubes and of pumps, which began shortly after NORCO began accepting waste fuel oil from vacuum tank trucks and tank cleaning operators, continued. A rotary twin screw pump formerly regarded as reasonably satisfactory became excessively worn after 60 hours of operation (about 100,000 gal.). The vanes in a rotary positive displacement pump lasted about 80 hours at the recommended speed of 500-420 RPM. The erosion in tubes appears to be most severe when the waste oil contains a high percentage of water. Resultant high velocities due to high steam formation cause severe erosion, especially when solids are present. Demulsification prior to processing to separate the bulk of the water present is very desirable.
1/15 - 2/15/73	The fractionator heater, transfer line, and fractionator were inspected. A leak at the west header of the first tube in the bottom radiant row was stopped by rerolling the tube end. A small leak resulting from erosion in the 4 inch by 6 inch elbow in the transfer line was found and repaired by welding a 3/8 inch thick steel patch over the heel of the elbow. A small amount of fine grit and coke was observed in the bottom of the fractionator, which probably accounts for the relatively quick erosion of the elbow in the transfer line.

12/7/72-1/15/73 The fractionator heater tubes and fractionator were thoroughly cleaned and repaired during December 1st to 15th, for the first time since March 20, 1972. Only four short crankcase oil runs have been made since that date. Two short drying runs were made drying part of the water out of the Berk's bottoms recovered from the lagoons at the Berk's plant. All other runs were made charging waste fuel oil. The fouling rate is probably one fifth as fast on waste fuel oil charged during this period as on crankcase waste oil. But very accurate determinations of fouling rates on waste fuel oil is difficult when runs on waste crankcase and Berk's bottoms are made between waste fuel oil runs.

One tube in the fractionator heater was replaced because of thinning from excessive metal temperature with resultant formation of iron oxide and sulfide scale on the outside of the tube. Allowable operating stack temperatures have been lowered to reduce the tube metal temperatures. The above will somewhat reduce the feed rate when processing oil. Seventy-two (72) stud bolts were re-welded on the heater tube headers. One tube was re-rolled in the headers. The 6" diameter section of the fractionator heater transfer line eroded through at a point of high turbulence just beyond the 6" x 4" steel elbow. A thick steel patch was welded over the eroded area. The fire brick of the combustion zone wall at the south side of the heater fell when high temperature refractory insulation moulded over supporting steel cracked and fell, exposing supporting steel to high temperatures and buckling. A new steel tee beam was installed and insulation replaced. The plastic refractory around the burner opening was replaced. The burner was cleaned.

11/7 - 12/7/72 NORCO's plant was designed primarily for crankcase waste oil, requiring no tank heating. However, with the onset of winter and increased acceptance of a wider range of all conceivable varieties of waste oils, which can be processed and blended to make saleable fuel oils, the need for extensive preheating is becoming apparent. This situation has been aggravated by the increased processing rate, starting with one 30 gpm feed pump then two 30 gpm feed pumps, and now with 50 and 30 gpm feed pumps installed. During the summer, piping was installed for using two 48,000 gallon tanks equipped with heaters as preheating means - also both tanks were equipped with air sparger piping arrangements for quickly and forcible agitating tank contents with short, large volume bursts of compressed air. Mixing results have been quick and effective. However, additional preheating will be required for handling of quantities of high pour and viscous oils at economic feed rates.

10/7 - 11/7/72 Since water does not settle rapidly from the more viscous oils, most of the water is removed in the fractionator. The limiting factor in processing wet oils has been the allowable pressure on the oil heater tubes and headers, and the heating capacity of the heater.

Determination of water content in many waste fuel oils cannot be satisfactorily obtained by the usual centrifuging tests, ASTM D1796 and ASTM D2709, because of formation of a gel which precipitates in the bottom of the centrifuge tube. Water in the oil apparently combines with various compounds in the oil to form the gel in a total volume which bears no direct relation to the volume of water. If the sample is diluted 1:1 with naphtha or kerosene and distilled in a manner somewhat like that described in the test ASTM D-95, the water content can be accurately and reliably determined. Modification of apparatus and procedure in distillation test ASTM D-95 provides a rugged inexpensive apparatus and satisfactory procedure for determining water content over a very wide percentage range. The sample may be heated and distilled to the required temperature to drive over all water much faster than when following the procedure specified in distillation test ASTM D-86. Also the dilution with naphtha or kerosene may be varied according to viscosity and probable water content, judged from gravity and appearance.

9/7 - 10/7/72 The oil heating coil in the flash tower fired heater ruptured during a crankcase oil run. It was patched for short time further service. This coil cannot be readily cleaned by the usual methods: steam-air decoking; chemical cleaning; or mechanical cleaning because of the nature of the deposits. A straight tube heater would be much preferred for this service.

8/7 - 9/7/72 During the month, sludge oil received from Pottstown (Berks) was processed primarily to remove water and trash. As received the oil contained considerable free water not combined as sludge or emulsion. It also contained considerable trash: leaves, twigs, sticks, dirt, grit, etc. The fluid to the unit varied abruptly from non-viscous practically 100% dirty water to 100% thick viscous emulsion. Control of flow and heating in the flow control instrumentation and tubular heater was erratic and difficult. Pressure, flow and temperature varied widely. The positive displacement charge pump wore rapidly. After about 100 hours of operation it was replaced with a Moyno pump, previously used for pumping oil and water mixtures from oil water separators. This pump successfully pumped the feed from the tank through about 250 ft. of 4" pipe and hose. The pipe was made up in 55'-60' sections connected with hoses. From NORCO's experience, general purpose waste oil plant operators would do well to develop a transportable and quickly connected system of pipe sections and hose sections for pumping all the various kinds of waste oils to be encountered. Readily transportable and quickly connected pumps with very short suction lines would complete the picture. For cold weather operation portable steam suction heaters and steam supply would be necessary. In some instances permanent equipment might be justified.

7/7 - 8/7/72 Wet waste oils from tank bottoms often are very high in specific gravity and pour point. When the water is evaporated from the oil the pour point of the dried fuel oil becomes "normal," that is low. This phenomenon may be associated with a "water-soluble waxy material" in these oils.

6/7 - 7/7/72 Steam piping size to the flash tower and fractionator heaters was increased from 1 inch to 1 1/2" to increase steam flow. The steam is important to the operation for: injection into the oil charge to maintain adequate velocity in the colder convection tubes of the fractionator heater; blowing oil out of the tubes at the end of runs; drying out of deposits in the inside of tubes before tube cleaning; and for injection into the heater fire box for snuffing out fires in case of tube failures. The deposit encountered from waste lubricating oil has quite different characteristics from that encountered in usual petroleum refining practice, tending to be much less brittle and frangible to impact of tube cleaner cutters. Flushing and drying increases frangibility and makes for faster satisfactory cleaning.

A 2 inch air line and hose were rigged up, to replace the existing one inch line, for blowing broken up deposits from the fractionator tube heater transfer line. Cleaning time was halved from 16 hours to 8 hours.

APPENDIX B
LOG OF RESEARCH AND DEVELOPMENT WORK

3/7-4/7/72	a. HRI laboratory report on hydrotreating. b. I-R spectrophotometry showed that the major portion of oxidized impurities in crankcase waste oil distillate cut had been reduced by hydrotreating. c. Ageing for 2 weeks at room temperature showed some additional deposit formation.
4/7-5/7/72	a. Solvent extraction of HRI hydrotreated oil improved color, haze, and odor to considerable degree.
5/7-6/7/72	a. Solvent extraction program continuing.
6/7-7/7/72	a. Examination of crankcase waste oil bottoms and sludge for Pb, Zn, Ba, S, Br, V showed Pb, Zn, Br in major quantities. b. Analysis of solids from filtration of tanker washdown effluent showed Zn, Cu, Fe, Mn, Pb, Br present in major quantities. c. Analysis showed low metals content in NORCO sidestreams #3 and #4. d. Laboratory centrifugation tests showed most metals showed up in recovered solids. e. Contacts made with organizations with possible interest in sludge. f. Solvents found which cause settling of aggregated polymer and solids. g. Wax and water contents determined for a waste oil.
7/7-8/7/72	a. Naphtha dilution followed by filtration upgraded Berk's bottoms (obtained as a result of hurricane caused spill in Schuykill Valley). b. Centrifugation of Berk's bottoms. c. Breaxit (Exxon) treatment of Berk's bottoms.

APPENDIX (continued)

7/7-8/7/72 (continued)	d.	Further solvent treating experiments.
	e.	Voges filter trial gave 3.5 gals/ft^2/hr. filtration rate.
8/7-9/7/72	a.	Bird and Centrico centrifuges were tried for solids recoveries from various waste oils and waste/oil mixtures.
9/7-10/7/72	a.	The possibility of effective filtration of waste oils was reviewed.
	b.	A bowl type Westphalia centrifuge was used to produce high lead solids. Solvent dilution produced a greater yield of solids.
10/7-11/7/72	a.	Modification of ASTM method for water determination for use with waste oils.
	b.	Further work on filtration of waste oils.
	c.	Determination of solids content of bottoms.
	d.	Solvent treatment of crankcase waste oils with methanol, ethanol, n-propanol, iso-propanol, n-butanol, iso-butanol, cyclohexanol, toluene, methyl ethyl ketone, acetone, amyl alcohol, 2-amino ethanol, glycerol.
11/7-12/7/72	a.	Further solvent treatment experiments using butanol, cyclohexanol, n-heptanol, hexanol, 2-furaldehyde, furfurol, dodecanol, tetraethylene pentamine, phenol, n-octanol, iso-octanol.
	b.	Solvent treating of Berk's bottoms with amyl alcohol with 10% pentane, methanol with 10% pentane, 50% methanol with 50% pentane, isopropanol with 10% pentane, amyl alcohol with 10% pentane, 50% butane diol with 50% methylethyl ketone.
12/7/72-1/15/73	a.	Further solvent treatment experiments on 2-amino ethanol, hexanol, heptanol, butanol.

APPENDIX (continued)

12/7/72-1/15/73 (continued)	b. Distillation of n-butanol/crankcase oil mixture. c. Separation of residue from NORCO bottoms. d. Iodine Number measurements made.
1/15-2/15/73	a. Separation of residue from NORCO bottoms and lead analysis. b. Separation of sludge and water from raw crankcase oil. c. Treatment of raw crankcase oil with 2-amino ethanol.
2/15-3/15/73	a. Preparation of high lead solids from crankcase oil tank bottoms. b. Crankcase and waste oil characterization. c. Phenol and n-butanol treatment of crankcase oil. d. Plant wastewater pH measurements.
3/15-4/15/73	a. Distillate bomb hydrogenation test. b. Crankcase and other waste oil characterization. c. Centrifugation tests on crankcase oil. d. Heat soaking/centrifugation cycle tests.
4/15-5/15/73	a. Crankcase and other waste oil characterization. b. Acetone and methanol treatment of crankcase oil.
5/15-6/15/73	a. Treatment of crankcase oil with tetraethyl ammonium hydroxide, diethylenetriamine, potassium hydroxide. b. Plant wastewater phenol and pH measurements.
6/15-7/15/73	a. Tests of Nalco demulsifying agents for fuel oil dewatering. b. Total acid number of raw crankcase oil and centrifuged crankcase oil. c. Chemical hydrogenation with borohydrides. d. Waste oil distillations.

APPENDIX (continued)

7/15-8/15/73	a. Chemical hydrogenation with borohydrides and lithium aluminum hydride. b. Waste oil characterization.
8/15-9/15/73	a. Chemical hydrogenation with borohydrides. b. Waste oil characterization. c. Ammonium hydroxide and heat treating of raw crankcase oil.
9/15-10/15/73	a. Chemical hydrogenation with borohydrides. b. Deodorization with KOH solution.
10/15-11/15/73	a. Chemical hydrogenation with borohydrides and sodium aluminum diethyldihydride. b. Deodorization with KOH solution. c. H_2SO_4 solution pretreatment.
11/15/73-3/15/74	a. Characterization of wastewaters b. Characterization of oil feeds and products.

APPENDIX C

CONSULTANT'S REPORTS ON WASTE OIL FILTRATION

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Weiss Laboratories
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September 29, 1972

Mr. Sol Maizus
Project Director
National Oil Recovery Corporation
P. O. Box 338
Bayonne, New Jersey 07002

Re: Contract No. 68-01-0177

Subject: Special Report on Filtration Procedures

Dear Mr. Maizus:

As per your request, I have prepared for you a comprehensive presentation of our efforts relating to this method of separating solid waste contaminants from oil.

We are omitting the obvious and obviously discarded methods such as plate and frame presses, intermittent paper filtration and organic membranes (for the filtration of submicron particles). The latter, because they are mainly used for biologicals, are far too/expensive and the flow rate is too/slow.

Our survey, therefore, extended over industrially suitable types including edge type filters which are capable of removing particles of five microns and up. Some of these systems require the replacement of the filter media upon use. Some are self cleaning by reverse blowing upon deposition of a filter cake.

An example of the former is offered by the Hilliard Corporation in Elmira, New York. The company manufactures a number of disc type filters using paper, rayon, fiberglass and asbestos combinations and stack mounted into tubular shaped elements. The latter self cleaning type is exemplified by Vokes Filter Division of The Cardwell Machine Company in Richmond, Virginia which is the American licensee of a British company.

A further group of filters is represented by Purolator and Cuno, both companies located in New Jersey.

The former company manufactures a wire wound filter element of cylindrical shape which can be cleaned (intermittently or continually by hand or automatically) by a peripherally attached scraping edge. In addition, Purolator manufactures a line of micronic resin impregnated paper filters as well as a thread wound filter cartridge known as micro-pac for every known fluid.

The latter company, namely, Cuno Division AMF, Inc., fabricates a metallic edge type filter composed of a stack of stationary discs interleaved by corresponding rotating wipers or cleaning blades.

In addition to the manual as well as the automatic version of this cleanable or self cleaning device, the company, Cuno, offers sintered metal filter elements, micro screen cartridges, asbestos and cellulose discs as well as pleated paper filter elements.

Both types of self cleaning filters described above exhibit element spacings of the disc stack or wire windings in the order of .001 to .003, thus setting a practical lower limit of 25 to 30 microns as the filter capability.

It seems clear from our investigations that the filtration of gelatinous media carrying sub-micronic occlusions presents a formidable problem to the separation effort. This is highlighted by the ultimate breakthrough of sub-micronic particles into a highly compressed paper filter disc after initial clear oil flow had been obtained. This was our experience with the Vokes filter which in all other respects is a superbly serviceable device. Once started, the sub-micronic contamination of the interior of a filter media will certainly preclude any further cleaning and continued plugging and micro-meritic bridging in the filter pores leads to ultimate clogging. It is, therefore, obvious that contamination must be held at the filter edge and it is clear that prior aggregation of the colloids as well as peptization of the gels is of utmost importance for the feasibility and economy of this separation procedure.

A further effort derived from the above considerations as well as our success in convincing EPA that the waste oil residue should not be burned but reclaimed and sold as a useful, valuable constituent containing lead and other metallic compounds. This residue is probably best produced in a continual process which precludes the necessity of batching and scraping of the putty-like substance (which is not readily ejected automatically from a centrifuge or filtration equipment).

Consequently, we evolve the notion of discarding the filter medium (paper) with the retained residue. Such a device known as the Trommel Vacuum Filter is made by Technical Fabricators in New Jersey. In it a newsprint type web is led over a drum which is connected to a vacuum pump. The drum is immersed in the fluid to be filtered and deposition is continuous and would permit the ensuing sandwich of filter paper and putty-like residue to be rolled up like logs, a very convenient form of packaging and transport indeed.

Our previous two progress reports recommended that we proceed to examine filtration as a practical means of separating our collected waste constituents.

Since the above mentioned Trommel Filter is solely operable by vacuum, we may incur some difficulty if we intend to use alcohol or other relatively low boiling diluents. One would have to resort to some form of cooling of the feed stock or preferably achieve a modification of this filtration method to allow for pressure rather than vacuum operation. Such a device would have to avail itself of a sealed chamber and we would be obliged to study the patent structure of the Trommel Filter to insure that no infringement exists.

Additionally, we have recommended that the use of filter aids or polyelectrolites such as Hercufloc be considered. The latter is organic and thus auto-degradeable in the following processing steps. Diatomaceous earth as well as clay could easily be used in small quantities and may even take the place of the more expensive chemical coagulants now being considered. The addition of a small amount of Dicalite or Filtros should add little of significance to the 26 elements already present in the lube stock in their various phosphate and calcium compound combinations. The subsequent reclamation steps conducted by the smelting companies must certainly include leaching, roasting or reduction steps capable of digesting such filter aids. It seems obvious that their use would materially influence the ease of deposition and filtration rates as well as costs. One might envision this as an adsorption-aggregation-filtration effect in a single step.

Beyond the above presentations, we have researched the efforts of separators for the treatment of industrial and sanitary waste. These include the Cata-Sep Oil/Water

Separators offered by the Pollution Control Division of PWI in Tulsa, Oklahoma, in addition to the SBS System produced in Santa Ana, California and waste water treatment stations utilizing tilted plate separators as manufactured by Cabinet F. Guiguos in France.

The Cata-Sep process utilizes in addition to a gravity separation system a unique catalytic cell to recover gasoline, fuel oil and other lube stocks from water. The catalytic cell is recleanable and is capable of separating varying amounts of oil from 20ppm to several percent from large volumes of water. This process will effectively break all oil/water emulsions but it is not clear from the literature submitted to us what effect oil wetted solids would have on the activity of this equipment.

However, this effect is not in doubt with the SBS System which utilizes what is essentially a fibrous resin bonded coagulating cartridge which is good for one half million to one million gallons at a flow rate of approximately ten gallons per minute. These cartridges are used in parallel multiples thus permitting the build up to any flow rate required. Their dimensions are as follows: 22" long by 6" in diameter and costing $27.50 per cartridge. A similarly dimensioned pre-filter cartridge which is recommended for use with waste containing large amounts of detergents or oil wetted solids sells for $14.50.

Weiss Laboratories
76-70 172nd ST., FLUSHING, N. Y. 11366 (212) 969-9794

November 30, 1972

Mr. Solfred Maizus
Project Director
National Oil Recovery Corporation
P. O. Box 338
Bayonne, New Jersey 07002

Re: Contract No. 68-01-0177

Subject: Monthly Progress Report #9
11/3/72 - 12/3/72

Dear Mr. Maizus:

This will apprize you of our progress during the preceding month to date:

1. The following represents an exhaustive treatment of filtration efforts to date and I think it also represents the state of the art as applied to our specific problems. To recap briefly: we were originally given a maximum flow rate of 2gph per square foot of filter surface by Technical Fabricators. This extremely low through-put prompted us to contact Tolhurst, a Division of Ametek, regarding perforated basket centrifuges. The thinking behind this move was primarily to provide much greater pressures than could be achieved by vacuum filtration and combined with the advantages of a disposable liner. Initial results with this equipment indicated a dramatic improvement which could, however, not be maintained for any length of time due to early blinding of the filter medium. The addition of diatomaceous earth resulted in a rate of 2gpm per square foot of filter surface and up to 6 gallons through-put before blinding occurred. The process, as contemplated, removes a 5 to 10 mil layer of the precoat per revolution. However, despite the satisfactory filtration rate, the disposal problem of over 10,000 lbs. of diatomaceous earth per day for full refinery

operation is considered unacceptable.

2. Since it was not clear whether the thickness of precoat which had to be "shaved off" was due to the difficulty of controlling a finer cut or because of sludge penetration depth, we once more swung over to Technical Fabricators with a request to precoat the newsprint filter medium used on the vacuum drum. By this device, we were able to increase the flow rate from the previous 2gph to 10-14gpm. In this effort the diatomaceous earth precoat was sprayed on the paper, but again filter aid use was considered to be excessive.

3. Up to this point all tests had been conducted with dilutions of Butanol in ratios of 2 to 1. In an effort to get rid of the filter aid, we decided to test some 4 to 1 dilution ratios and found that a batch, which had been prepared some weeks ago, now yielded considerably reduced filtration rates than had been obtained when this mixture was freshly prepared. We, therefore, made up another quantity of 5 gallons consisting of 4 parts Butanol and 1 part lube oil stock. To one-half of this quantity (2-1/2 gallons) was added 300c.c. of Pentane in an effort to produce a "drier", i.e., less gelatinous deposit. This freshly prepared material was filtered without filter aid at the rate of 17gph for the part containing Pentane and 20gph for the pure Butanol mixture.

4. Considering all factors such as equipment costs, based on processing speeds as well as material expense and disposal problems, this represents probably the optimum result to date. At the drum velocity of 1rpm, we would consume approximately $40.00 worth of paper per hour when processing 400,000 gallons per day. The paper is, of course, consumed in the reclamation of the metals from the sediment and presents no disposal problem whatsoever.

5. One additional alternative is contained in the proposal by Ametek to conduct further tests at

their East Moline, Illinois laboratory using Solka-Floc in place of inorganic filter aids. This substitution would also eliminate the disposal problem and quite probably result in further increased through-put rates.

COMMENTARY

This work was conducted with specific emphasis on continuous operation as compared to the intermittent removal of centrifuge sludge. As we pointed out previously, our filtrate is an unusually difficult combination of small and sub-micron particles in gelatinous media and is thus extremely hard to separate. Additionally, the material exhibits a tacky consistency thus complicating its removal from equipment surfaces even further. For those reasons, we have concentrated on the ablative techniques described above and feel that similar techniques might be investigated in connection with the distillation equipment used.

Very truly yours,

WEISS LABORATORIES

GERHART WEISS

GW/ar

RESULTS OF CENTRIFUGATION TESTS USING A BOWL TYPE CENTRIFUGE

Crankcase oil

The feed sample of crankcase oil when analyzed by the high speed "Sorvall" centrifuge gave a sludge value of 3.8% by weight. The effluent of the chamber type clarifier, when run under a flow rate of 100 gal./hr., gave a value of 2.7% sludge. The efficiency of the clarifier was therefore an approximate 29% when running at 8100 G and residence time of approximately 10 minutes.

The accumulation of solids product varied from 19 lbs./ 1000 gal. run to 13.3 lbs./1000 gal. run. Analysis showed 13.5% lead content.

Crankcase oil treated with solvent (n-butanol)

The crankcase oil was treated with solvents at the ratio of 1:1 and 1:2. Since agglomeration occurs with solvent, the analysis of input feed is critically dependent on sample taken. Because of the time delay in workup and transmission of proper samples, a great variability was found in the sample workup. A typical effluent using an oil/solvent ratio of 1:2 yielded a 0.07% solids content. The actual run using a 1:2 ratio gave 10 lbs., of sludge per 100 gal. of oil, and with a ratio of 1:1 yielded 7 lbs. of sludge per 100 gals. of oil. The efficiency of agglomeration is evident with the solvent as compared with straight crankcase oil (1.9 to 1.3 lbs. of sludge per 100 gals. oil) and with crankcase oil diluted 1:1 with naphtha.

Analysis of the solids obtained from a 1:2 centrifugation run yielded 15.9% lead.

Berks Oil

100 gals. of Berks waste oil bottoms were centrifuged as obtained and 23 lbs. of solids were collected. Analysis of this solid for lead showed an 18.2% content, a value which is of interest to metals re-refiners.

Coal Tar Oil

100 gals. of coal tar oil pumped from #105 tank was fed to the centrifuge. Solids buildup was somewhere between 20 and 25 lbs.

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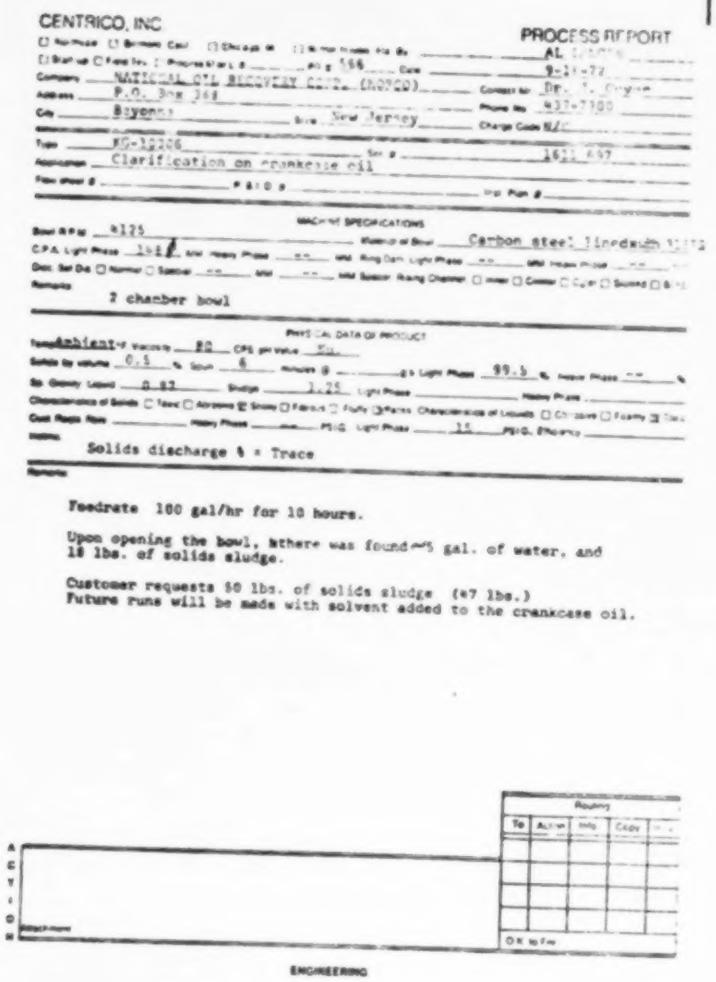


CENTRICO, INC

PROCESS REPORT

AL [illegible]

PR # 566

Date 9-1[illegible]-72

Company NATIONAL OIL RECOVERY CORP. (NORCO)

Contact Dr. [illegible]

Address P.O. Box 368

City Bayonne

State New Jersey

Charge Code N/C

Type [illegible]-12206

Ser # 1621 [illegible]

Application Clarification on crankcase oil

MACHINE SPECIFICATIONS

Bowl RPM 4125

Material of Bowl Carbon steel [illegible]

C.P.A. Light Phase 161 MM Heavy Phase -- MM Ring Dam Light Phase -- MM Heavy Phase --

Remarks

2 chamber bowl

PHYSICAL DATA OF PRODUCT

Temp Ambient °F Viscosity 80 CPS pH Value [illegible]

Solids by volume 0.5 % Spun 6 minutes @ ____ Light Phase 99.5 % Heavy Phase -- %

Sp. Gravity Liquid 0.87 Sludge 1.25

Light Phase 15 PSIG

Solids discharge % = Trace

Remarks

Feedrate 100 gal/hr for 10 hours.

Upon opening the bowl, there was found ~5 gal. of water, and 18 lbs. of solids sludge.

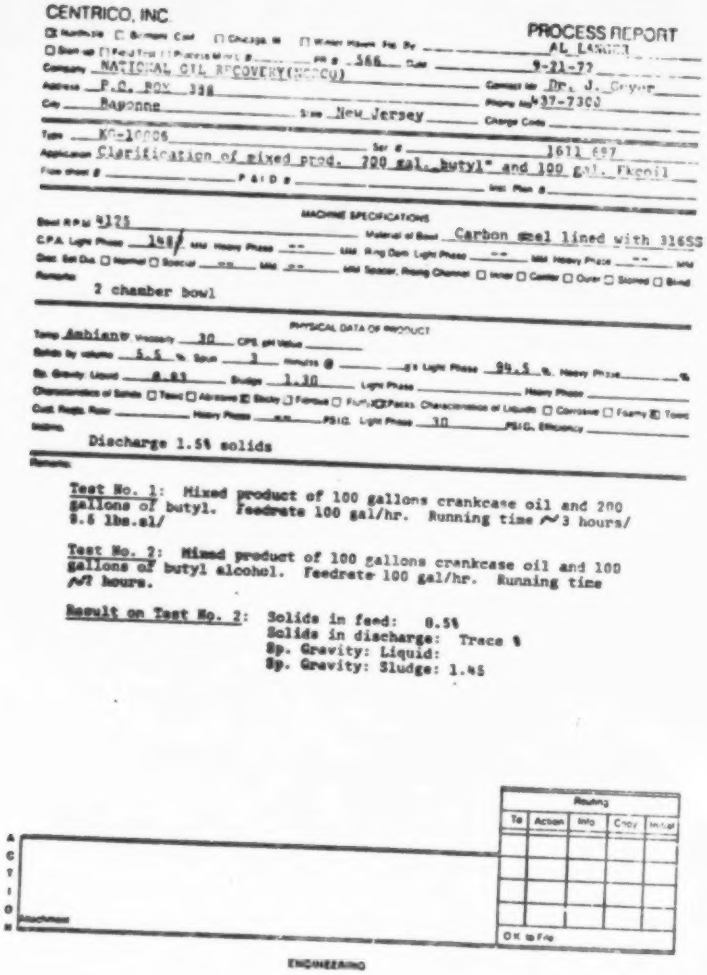
Customer requests 50 lbs. of solids sludge (47 lbs.)
Future runs will be made with solvent added to the crankcase oil.

Routing				
To	Action	Info	Copy	
O.K. to File				

ACTION

Attachment

ENGINEERING



CENTRICO, INC.

PROCESS REPORT

☒ Northvale ☐ Bergen Cnty ☐ Chicago, Ill. ☐ Winter Haven, Fla. By AL LANGER
☐ Start up ☐ Field Test ☐ Process Mont. # PR # 566 Date 9-21-77
Company NATIONAL OIL RECOVERY(N[illegible]CO)
Address P.O. BOX 338 Contact Dr. J. Geyer
City Bayonne State New Jersey Phone No. 437-7303
Charge Code
Type KG-10006 Ser # 1611 697
Application Clarification of mixed prod. 200 gal. butyl" and 100 gal. Fkcoil
Flow sheet # P & I D # Inst. Plan #

MACHINE SPECIFICATIONS

Bowl RPM 4125
C.P.A. Light Phase 148 MM Heavy Phase -- MM Material of Bowl Carbon steel lined with 316SS
Ring Dam Light Phase -- MM Heavy Phase -- MM
Disc. Set Dia. ☐ Normal ☐ Special -- MM -- MM Spacer, Rising Channel ☐ Inner ☐ Center ☐ Outer ☐ Slotted ☐ Blind
Remarks:
2 chamber bowl

PHYSICAL DATA OF PRODUCT

Temp Ambient Viscosity 30 CPS pH Value
Solids by volume 5.5 % Spun 3 minutes @ G's Light Phase 94.5 % Heavy Phase %
Sp. Gravity Liquid 0.83 Sludge 1.30 Light Phase Heavy Phase
Characteristics of Solids ☐ Toxic ☐ Abrasive ☒ Sticky ☐ Fibrous ☐ Fluffy ☒ Packs Characteristics of Liquids ☐ Corrosive ☐ Foamy ☒ Toxic
Cont. Reqd. Rate Heavy Phase n.a. PSIG. Light Phase 30 PSIG. Efficiency
Instrns.
Discharge 1.5% solids

Remarks:

Test No. 1: Mixed product of 100 gallons crankcase oil and 200 gallons of butyl. Feedrate 100 gal/hr. Running time ~3 hours/ 8.6 lbs.sl/

Test No. 2: Mixed product of 100 gallons crankcase oil and 100 gallons of butyl alcohol. Feedrate 100 gal/hr. Running time ~2 hours.

Result on Test No. 2: Solids in feed: 0.5%
Solids in discharge: Trace %
Sp. Gravity: Liquid:
Sp. Gravity: Sludge: 1.45

ACTION

Attachment

Routing				
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ENGINEERING

[illegible]

CENTRICO, INC.

January 11, 1973

Mr. N. J. Weinstein
RECON SYSTEMS, INC.
Cherry Valley Road
Princeton, New Jersey 08540

Dear Mr. Weinstein:

Enclosed please find a copy of our general catalog as well as the separate sheet covering the KO-10006 which is basically the same machine which was run at National Oil Recovery on your products. The 2 basic types of machines which would be applicable for this project would be the desludger type and the chamber bowl type, both of which are described in the bulletin. It is in our opinion doubtful if the desludger type would be able to consistently discharge the hard packing solids completely and therefore at the present time we would not recommend such a machine.

The chamber type bowl KG-10006 has a maximum G force of 6000. The approximate price for such a machine would be $18,000, including the motor. Strictly for comparison's sake, a desludger of approximately the same size would have a price of roughly $40,000.

We hope the enclosed information will aid you in determining the feasibility of applying centrifugal gears toward a solution of your problem. It is CENTRICO'S feeling that this is not an ideal application and unless the economic side of it looks extremely good, we do not feel that our equipment is applicable.

If you should have any further questions or if you would like to visit with us and discuss the project we would be happy to do our best to assist you at any time.

Very truly yours,

CENTRICO, INC.

V. Lindenman
Process Engineer

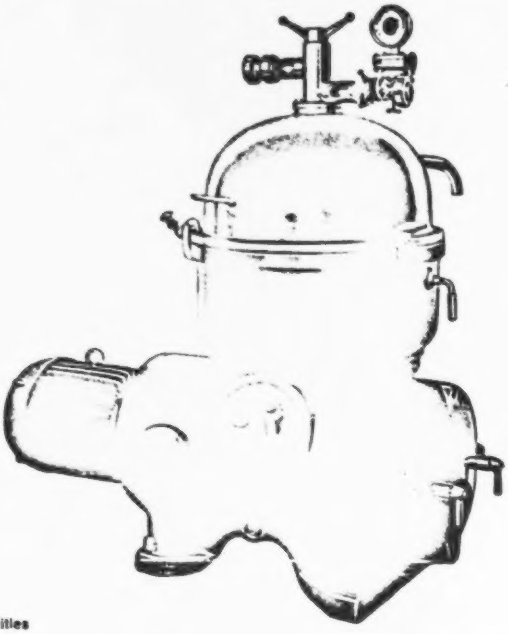
Our Ref.- PR# 566

CENTRIFUGAL SEPARATORS CLARIFIERS CONCENTRATORS

Chamber-type Bowl Clarifier Model KO 10006

(Supplementary leaflet to KO Pamphlet 3545/667)

Since 1969 WESTFALIA SEPARATOR AG has extended the KO range of clarifiers to include model KO 10006 in addition to models KO 2006 and KO 8006. The previous type KG 10006 has thus been replaced by a more modern version of the same capacity. Particularly the fittings of the KO range are arranged in a clearer fashion than those of the old KG type clarifiers. The fittings can be easily removed together with the hood permitting quick access to the bowl.



Technical data and capacities

Model			KO 10006	
Two-chamber bowl	total capacity		litres	76.5
	sludge holding capacity		litres	65
Six-chamber bowl	total capacity		litres	75
	sludge holding capacity		litres	60
Motor power			kW	11
			hp	15
Bowl speed*			rpm	4500
Capacities**	Hot beer wort	up to	litres/h	10000
			Imp. gals./h	2200
			US. gals./h	2640
	Fruit juice	up to	litres/h	3000
			Imp. gals./h	660
			US. gals./h	790
	Red wine	up to	litres/h	5600
			Imp. gals./h	1230
			US. gals./h	1480
Capacities**	Molasses	up to	kg/h	3000
			lbs/h	6600
	Hot clear varnishes	up to	kg/h	5600
			lbs/h	12300
	Liquors	up to	litres/h	11000
			Imp. gals./h	2400
			US. gals /h	2900

Weight of complete machine with motor		net kg	1130
		net lbs	2500
		gross kg	1370
		gross lbs	3000
Packing case dimensions	frame with motor	length in cm	166
		in inches	65
		width in cm	96
		in inches	38
		height in cm	125
		in inches	50
Packing case dimensions	bowl	length in cm	71
		in inches	28
		width in cm	71
		in inches	28
		height in cm	70
		in inches	27
Weight of bowl		net kg	400
		net lbs	1000
		gross kg	460
		gross lbs	1010

Clarifier models KO 2006, KO 8006, and KO 10006 meet the requirements of the Federal German Physical and Technical Institute (PTB) and may be used wherever explosion hazards exist.

---

* The speed depends on the specific gravity of the feedstock and on the bowl material used. It may therefore differ from the normal speed given above.

** The hourly capacities depend on viscosity, temperature, difference in specific gravities of feedstock and solid matter, character of the solids to be removed and the desired degree of purity of the clarified liquid. If the particle size of the solids is very small and there is little difference between the specific gravities of solids and liquid, the hourly capacity must be reduced to extend the retention time in the bowl. The maximum throughput capacity of the bowl is, however, considerably higher than indicated.

APPENDIX E

HYDROTREATING ANALYSES AND DATA

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CATALYTIC HYDROTREATING EXPERIMENTS

SUMMARY

A sample of combined sidestreams obtained from the vacuum distillation in the National Oil Recovery Corporation's plant was hydrotreated by Hydrocarbon Research, Inc. The color improved from 8 ASTM to 3 ASTM, and the API gravity increased by two points. The color and odor were excellent and it was indicated that, even after two days standing at room temperature, the color was maintained at this high level.

In addition, the Hydrocarbon Research Laboratory performed a vacuum distillation on a full waste crankcase oil sample and then charged this sample immediately to their hydrotreating facilities in the pilot plant laboratory. The vacuum distillation was operated so as to cut into the waste crankcase to the extent of allowing the production of only ten percent bottoms. This sample was indicated to be darker in color as feed to the hydrotreater than the previous NORCO distilled sample; however, the product after hydrotreating was of higher color quality than the previous sample, and the odor was identical.

There were no operability limitations or any indication of undue plugging of the catalyst. The pressure was 600 pounds per square inch at 700°F.

PRELIMINARY NORCO WASTE LUBE OIL HYDROTREATING STUDY

Report By

W. C. Rovesti
R. H. Wolk

Hydrocarbon Research, Inc.

Laboratory Report Number L-1236-501

April 18, 1972

INTRODUCTION

The successful reclamation of waste crankcase oil is important both for the conservation of our natural resources and for ecological considerations. In order to reduce a suspected tendency to dispose of the waste oil in a manner which is detrimental to the environment; e.g., dumping on the ground, into sewers or a convenient waterway, it is desirable to be able to convert this material into saleable products. Simply burning the waste oil, in addition to heavy fouling of combustion equipment, results in the release of its heavy metal contaminants into the atmosphere where they could become a serious air pollution problem.

The National Oil Recovery Corporation (NORCO) waste oil re-refinery in Bayonne, New Jersey recovers three side streams from a vacuum distillation tower: a light fraction, diesel blending components, and fuel oil. All of these are dark in color and contain varying amounts of tarry components. A blend of light and heavy side streams from the vacuum distillation was found to contain small amounts of a solid residue which is thought to form upon standing. These polymeric solids form a rather stable "floc", thereby causing the product to have an undesirable color. The persistance of an offensive odor also detracts from the salability of this product.

In order to study means by which the quality of this blend of the light and heavy side streams may be improved, NORCO has commissioned HRI to carry out a short term study to determine the effect of hydrotreating on the product quality. The intent of this study is to provide preliminary data rather than to establish optimum operating conditions and catalyst or provide data on catalyst deactivation, etc.

SUMMARY AND CONCLUSIONS

A preliminary fixed-bed hydrogenation study was performed on a blend of side streams from the vacuum distillation of waste crankcase oil carried out at NORCO's Bayonne, New Jersey re-refinery. Freshly distilled heavy vacuum overhead prepared at HRI was also hydrotreated as part of the study. The laboratory distillation was carried to a higher end point, and this yielded a higher proportion of distillate and a substantially lower quantity of vacuum bottoms. The object of this work was to determine the effect of hydrotreating on product quality, especially color, color stability, odor and metals contaminants.

Based on the results obtained during the course of the study, the following can be concluded about the effect of hydrotreating on product quality.

1) A substantial improvement in color and color stability for the side stream blend provided by NORCO.

2) An even greater improvement in color, which was stable, was obtained for freshly distilled feed.

3) Removal of offensive odor found for both feeds.

4) Apparent reduction in lead (the major contaminant) content by well over an order of magnitude for both feeds.

In addition, no problems with the operability of the fixed bed hydrotreater were encountered after a total of 67 hours on stream using both the NORCO blend and the freshly distilled feed.

In summary, the results of this study indicate that hydrotreating may be a very effective way of improving the product quality and salability of products from the reclamation of waste crankcase oil. The positive results achieved during this test program indicate that additional laboratory and engineering work is justified in order to arrive at an optimum processing scheme.

EXPERIMENTAL

Feed Materials

Three liquid samples from NORCO were received on March 21, 1972. These were labeled waste crankcase oil (HRI 3343), light side stream (HRI 3344), and heavy side stream (HRI 3345). A representative sample of the waste crankcase oil was taken for analyses. However, no analyses* of the light or heavy side streams were made. All analytical results are presented in Table 1.

A blend (L-322) of three volumes of the light cut (HRI 3344) and two volumes of the heavy cut (HRI 3345) was prepared. This preparation was reported to be the same as that produced in the NORCO vacuum distillation and constitured 65-volume percent of the distilled product. Approximately 20-volume percent bottoms and 15-volume percent light overhead were produced in the plant distillation. Analysis of the blend is also presented in Table 1.

---

* NORCO reports a gravity of 31 to 31.5°API for the light cut and 29.5 to 29.7°API for the heavy cut.

and the 99 V 5 end point was 940°F.

Fixed Bed Operation

The NORCO side stream blend (L-322), and subsequently, freshly prepared heavy vacuum overhead from the distillation of the waste crankcase oil, were hydrotreated in a continuous bench-scale, fixed bed pilot unit. A once-through operation with no recycling of either liquid or gas was employed. The catalyst used was a commercially available 1/16" extrudate hydrotreating catalyst. The catalyst was presulfided prior to use. Operating conditions employed were:

Hydrogen Pressure - 600 psig
Temperature - 700°F
Hydrogen Gas Rate - 1500 SCFB
Liquid Volume Space Velocity - 1.0 $V_o/hr/V_c$
Catalyst*

The hydrotreating operation (Run 184-131) was begun with the NORCO side stream blend and after a total of 44 hours of continuous operation, the heavy vacuum overhead from the first batch distillation was fed to the unit. Subsequent distillations were carried out to provide sufficient feed. The heavy vacuum overhead was hydrotreated for 23 hours before a voluntary shutdown of the unit was made.

---

*American Cynamid HDS-3A Nickel/Moly 1/16" extrudate

Vacuum Distillation of Waste Crankcase Oil

Vacuum distillation of the total waste crankcase oil (HRI 3343) was carried out in order to study the effect of hydrotreating on a freshly prepared intermediate fraction distilled under conditions where a minimum of 10-volume percent bottoms are produced. The distillation was carried out in a batch still (Unit 200). A charge to the still was approximately 3500 grams. Several distillations were made in order to provide sufficient feeds for the hydrotreating run, described later in this section.

A sample of the heavy vacuum gas oil (L-323) which was fed to the fixed bed unit during the 8-hour test period (184-131-Period 3D) was analyzed. The analytical results are presented in Table I. The vacuum distillation employed in the preparation of L-323 was carried out to give the following cuts:

Fraction	Weight %
Knock Outs Plus Light Vacuum Overhead	18.3
Heavy Vacuum Overhead (L-323)	76.6
Vacuum Still Bottoms	5.1

The charge (L-323) to the fixed bed hydrotreater (Unit 184) during Period 3D had gravity of 29.8°API compared to 30.5°API for the NORCO side stream blend (L-322). The IBP of L-323 was 492°F

RESULTS

Operability

A total of 67 hours of trouble-free operation were carried out using the two feeds. There was no apparent difference between the NORCO blend (L-322) and the freshly distilled heavy vacuum overhead (L-323) from the standpoint of operability over the short-term, fixed bed hydrogenation run.

Analytical Results

The analyses carried out on the total waste crankcase oil (HRI 3343), the blend of NORCO side streams (L-322), its hydrotreated product (184-131-1B), the freshly distilled heavy vacuum overhead (L-323), and its Hydrotreated product (184-131-3D) are summarized in Table 1.

The metal analyses were run using atomic absorption spectroscopy. Both hydrotreated products contained a small amount of dark particulate matter which readily settled to the bottom of their containers. The samples were decanted to avoid possible blockage of the atomic absorption unit burner. Therefore, the metal analyses of the products do not reflect any metals which may be in the settled particulates.

The ASTM color was run according to the ASTM standard D1500-64. Hydrotreated products were purged with nitrogen in order to remove the dissolved H_2S prior to running the determination.

Product Odor

Both products were "sniffed" after being purged with N_2 to remove dissolved H_2S. The odor characteristic of the feeds appeared to be absent.

TABLE 1

SUMMARY OF ANALYSES FROM PRELIMINARY NORCO HYDROTREATING STUDY

	Total Waste Crankcase Oil HRI 3343	NORCO Side Streams Blend L-322	Hydrotreated Product (From L-322) 184-131-18	HRI Vac. Distill. Of HRI 3343 L-323	Hydrotreated Product (From L-323) [illegible]
W % S	0.22	<0.07	<0.07	0.31	<0.07
°API	25.2	30.5	32.3	29.8	31.9
V % (IBP-400°F)	----	----	<2	----	<3
V % (IBP-600°F)	----	----	8	----	5
N, ppm	804	256	79	327	[illegible]
Pb, ppm **	----	2	0.05	11	0.12
Ca, ppm **	----	0.25	0.05	0.02	0.02
Na, ppm **	----	N.D.	N.D.	N.D.	N.D.
Fe, ppm **	----	0.37	0.97	0.74	0.84
Wt% Ash	1.41	----	----	----	----

ASTM, COLOR					
Initial	D8	L7.5	L3.5	L7.5	[illegible]
Δ t = 4 Hours	----	----	L3.5	L7.5	[illegible]
Δ t = 48 Hours	----	L7.5	L3.5	L7.5	[illegible]
Δ t = 96 Hours	----	----	L3.5	L7.5	L2.5

VACUUM DISTILLATION (°F) 1.2 MM					
IBP	<405	408	418	492	<465
5 V %	----	598	483	640	----
10 V %	585	(650*)	545	676	624
20 V %	692	672	641	715	[illegible]
30 V %	730	698	670	741	716
40 V %	755	728	704	779	742
50 V %	786	756	741	795	[illegible]
60 V %	815	778	755	804	[illegible]
70 V %	849	796	772	835	827
80 V %	892	815	790	850	[illegible]
90 V %	945 (Cracked)	841	825	880	[illegible]
95 V %	----	----	856	897	----
99 V % E.P.	----	925	885	940	[illegible]

N.D. = None Detected
* = Denotes temperature at 12.5 V %
** = Samples decanted to avoid getting settled particulates into A.A. burner.

March 7, 1973

Mr. Solfred Maizus
President,
NATIONAL OIL RECOVERY CORPORATION
P. O. Box 338
Bayonne, New Jersey 07002

Dear Sol:

I have drawn the following observations and conclusions from my inspection and analysis of the Exxon data and other data available on distillate hydrotreating:

1. Hydrotreating a lube oil distillate obtained by vacuum distillation of crude waste oils can provide a product whose properties are similar to a typical 150 Vis Neutral Lube Base Stock, including satisfactory color, color stability, carbon residue, and corrosion properties.

2. Distillates from at least five different waste oils have been hydrogen treated effectively; two different catalysts have been used: and temperatures have ranged from 550°F. to 700°F.

3. In the longest continuous operation, about 100 hours was logged with no noticeable catalyst deactivation.

4. Hydrogen consumption data was not obtained in any of the work reported.

5. From theoretical calculations based on feed and product properties, I expect that hydrogen consumption may be close to 150 SCF per barrel of distillate, costing about 1¢/gallon of raw feed (for hydrogen purchased at $4.00 per 1000 SCF).

6. Except for NORCO's own work, no information is available on corrosion, fouling, product performance as a lubricating oil, or methods for metals recovery.

COPY

In order to firm up the technical basis for completing the development of the vacuum distillation/hydrogen treating process, I recommend:

1. Batch bomb tests in NORCO's laboratory for preliminary hydrogen consumption measurements. Dr. Geyer and I are already arranging such tests.

2. Flow experiments to be conducted in an outside laboratory to firm up hydrogen consumption. I have discussed such experiments with Sun Oil Co. This should involve about two weeks or less work, if hydrogen consumption is reasonable. The experimental period may be lengthened if a parameter study is required to minimize hydrogen consumption while making satisfactory product. It is also proposed that the Sun work include certain performance capability tests, as outlined in their letter of February 16, 1973. HRI will also be contacted to ascertain their interest and ability to carry out this work.

3. A life test on the catalyst of choice. In view of the favorable data to date, I consider this test to be non-critical and plan to delay initiation until the above work is complete.

4. Furnace design studies to provide a basis for minimizing fouling problems. This should be started shortly if we are to meet our previous schedule. I hope to discuss this with you within the next week.

5. Continuing research on metals recovery from the distillation bottoms product.

COPY

I have attached some of my most pertinent calculations and data summaries for your use.

Sincerely,

Norman J. Weinstein
President

NJW/gtf
Enclosures

cc: Messrs. R. Keppler
J. Geyer
K. Urquhart

LUBE OIL DISTILLATE HYDROTREATING

PREDICTION OF HYDROGEN CONSUMPTION

Basis: Exxon Data
$650^{\circ}F.$, 650 psig., 1 V/V/Hr., 800 SCF/B

	HYDROGEN CONSUMPTION SCF/B DISTILLATE
Aromatic Saturation	110
Desulfurization	5
Denitrogenation	3
Deoxygenation	30
Olefin Saturation	5
	153 SCF/B
	or 3.64 SCF/Gal.

COPY

For 0.4¢/SCF H_2, Cost = 1.46¢/Gal. Distillate

For 0.64 Gal. Distillate/Gal. Raw Waste Oil,
Cost = 0.94¢/Gal. Raw Waste Oil*

This compares with 0.09¢/gal. Raw Waste Oil used in original economic study.

* This is essentially Case 9, Table 3 of the "Report to NORCO on the Potential Profitability of Several Approaches to Re-Refining Crankcase Waste Oil," N. J. Weinstein, January 22, 1973.

RECON SYSTEMS, INC.
Princeton, N. J.
N. J. Weinstein
March 7, 1973

LUBE OIL DISTILLATE HYDROTREATING

MAXIMUM HYDROGEN CONSUMPTION CALCULATIONS

	Distillate Properties	
Gravity, °API	29.8	
Sulfur, Wt. %	0.31	
Nitrogen, Wt. %	0.04	
Oxygen, Wt. %	0.35	
Hydrogen, Wt. %	13.08	(from gravity correlation)
Iodine No.	1.0	

	Maximum Hydrogen Consumption SCF/B	
$S \rightarrow H_2S$	11.3	
$N \rightarrow NH_3$	5.0	
$R_1R_2C = O \rightarrow R_1R_2CH_2 + H_2O$	50.9	
$R_1HC = CHR_2 \rightarrow R_1 (CH_2)_2 R_2$	4.6	
Aromatics → Naphthenes	871.0	(Based on 2 H atoms/C atom)
	942.8 SCF/B = 22.5 SCF/Gal.	

COPY

For 0.4¢/SCF H_2, Max. Cost = 9.0¢/Gal. Distillate Feed

or 0.64 x 9.0 = 5.76¢/Gal. Raw Feed for 64% Yield

RECON SYSTEMS, INC.
Princeton, N. J.
N. J. Weinstein
March 7, 1973

LUBE OIL DISTILLATE HYDROTREATING
CALCULATED HYDROGEN CONSUMPTION
BASED ON HYDROTREATING EXPERIMENTS

	EXXON (4) 650 psig., 1 V/V/Hr., 800 SCF/B			URI (7) - 600 PSIG 1 V/V/Hr., 1500 SCF/B		IFP-Lube Finishing (11)	
Temp., °F.	550	600	650	700	700	?	?
Gravity, °API							
Feed	30.7	30.7	30.7	30.5	29.8	27.0	24.7
Product	31.3	31.4	31.5	32.3	31.9	28.5	25.6
A. Wt. % H_2(1)							
Feed	13.23	13.23	13.23	13.20	13.07	12.50	12.04
Product	13.36	13.38	13.40	13.52	13.46	12.80	12.23
	0.13	0.15	0.17	0.32	0.39	0.30	0.19
B. Wt. % S							
Feed	0.12	0.12	0.12	<0.07	0.31	1.5	2.0
Product	0.053	0.031	0.012	<0.07	<0.07	0.6	0.8
	0.067	0.089	0.108	---	<0.31	0.9	1.2
C. Wt. % N							
Feed	0.018	0.018	0.018	0.0256	0.0327	?	?
Product	0.006	0.005	0.002	0.0079	0.0022	?	?
	0.012	0.013	0.016	0.0177	0.0305		
D. Wt. % O							
Feed	?	?	?	?	?	--	--
Product	?	?	?	?	?	--	--
Calc H_2 Consumption, SCF/B							
A	98.2	103.9	109.8	219	255	194.4	137.7
B	2.4	3.2	3.9	---	<11.2	33.3	45.1
C	1.5	1.6	2.0	2.2	3.8	?	?
D	?	?	?	?	?	--	--
Olefin Sat.	?	?	?	?	?	--	--
Total	102.1+	108.7+	115.7+	221.2+	270.0+	227.7	182.8
Reported	--	--	--	--	--	140	185

OIL INSPECTIONS
RANGES ENCOUNTERED

	RAW WASTE LUBE OIL	VACUUM TOWER DISTILLATE(3)	HYDRO-TREATED DISTILLATE	TYPICAL 150 VIS NEUTRAL LUBE STOCK	RE-REFINED OILS (8,9)
Gravity, °API	22.0-27.9 (2,4,6)	29.8-30.7 (4,7)	31.3-31.5 (4)	31.8 (4)	24.6-29.0
Visc. @ 100°F.,SSU	137-549 (2,4,6)	144-197.9 (4)	156-162 (4)	157 (4)	271-930
Visc. @ 210°F.,SSU	55.4-95.8 (4,6)	43.1-46.5 (4)	43.8-44.1 (4)	43.9 (4)	53.5-75.9
Viscosity Index	127-196 (4)	104-105 (4)	101-104 (4)	104 (4)	88-105
Flash Point, °F.	175-400 (2,4,6)		410-430 (4)	415 (4)	
Pour Point, °F.	(-30)-(-45) (4)		(+15)-(+20) (4)	+15 (4)	
Neutr. No.	4.3-7.3 (4)	0.23-0.59 (4)	0-0.005 (4)	0.01 (4)	0-0.19
CCR, Wt. %	3.3-12.6 (2,4)		0.001 (4)	0.01 (4)	0.23 (9)
Copper Strip Corr.	-		1-2 (4)	1 (4)	
Ash, Wt. %	1.0-3.78 (2,6,7)				Trace (9)
Sulfated Ash, Wt.%	1.02-2.41 (6)				
Water, Vol. %	0.05-11.0 (1,4,6)				
BS&W, Vol. %	Approx. 2.4-15 (6)				
Sapon. No.	11.3-35.4				
Pentane Insol., Wt. %	1.5-7.3 (1,6)				0
Sulfur, Wt. %	0.21-0.34 (4,6,7)	0.07-0.31 (4,7)	0.012-0.07 (4,7)	0.08 (4)	
Nitrogen, Wt. %	0.08-0.21 (4,7)	0.02-0.04 (4,7)	0.002-0.008 (4,7)		
Oxygen, Wt., %	1.36-2.25 (4)	0.16-0.35 (4)			

OIL INSPECTIONS (Continued)

	RAW WASTE LUBE OIL	VACUUM TOWER DISTILLATE(3)	HYDRO-TREATED DISTILLATE	TYPICAL 150 VIS NEUTRAL LUBE STOCK	RE-REFINED OILS
Phosphorous, Wt.%	0.055-0.11 (6)				0.001
Color, ASTM	Black (4)	3.5-8.0 (4,7)	0.5-3.5 (4,7)	1.5 (4)	3½-7 NPA
Color Stability	-	-	16-18 (4,5); L2.5-3.5 (7,12)		
Metals, ppm					
Lead	2000-22,000 (4,6,10)	0.5-11 (7,10)	0.05-0.4 (7,10)		1 (9)
Zinc	350-980 (4,6,10)	0.4-0.5 (10)	0.3 (10)		0.5 (9)
Barium	100-1000 (6,10)	<0.01 (10)	<0.05 (10)		0.5 (9)
Calcium	700-2100 (6,10)	0.02-0.25 (7,10)	0.02-0.3 (7,10)		1 (9)
Iron	53-2000 (6,10)	0.4-1.9 (7,10)	0.17-0.97 (7,10)		
Fluoride	36 (10)	12 (10)	8-9 (10)		
Other Elements (>1ppm)	Mg,V,B,Cd,Cr, Cu,Mn,Ni,Sn, K,Si,Na,Sr,Mo, Ti, (6,10)	B,Sn,Si	None		

RECON SYSTEMS, INC.
Princeton, N. J.
M. J. Weinstein
March 7, 1973

REFERENCES and FOOTNOTES

1. Environmental Quality Systems, Inc. (EQSI), "Waste Oil Recovery Practices - State-of-the-Art," December 1972, Table 8B.

2. EQSI, op.cit., Table 8C.

3. Approximately 400-1050°F. boiling range.

4. Bethea, S. R., et al., "A Modern Technique for Automotive Waste Oil Re-Refining: Distillation + Hydrofining," Letter from G. R. L. Shepherd, Esso Research and Engineering Co. to P. B. Lederman, U. S. Environmental Protection Agency, March 1, 1973.

5. Tag-Robinson color after 16 hours at 212°F.

6. API, "Final Report of the Task Force on Used Oil Disposal," Publication No. 4036, 1969.

7. Rovesti, W. C. and Wolk, R. H., "Preliminary NORCO Waste Lube Oil Hydrotreating Study," HRI Lab. Report No. L-1236-501, April 18, 1973.

8. Armour Research Foundation, "Study of Re-Refining Waste Disposal," Reports to Association of Petroleum Re-Refiners, 1960-62.

9. Bonnifay, P., et al. (IFP), "A New Process for Reclaiming Spent Lubricating Oils," presented at the National Fuels and Lubricants Meeting, September 14-15, 1972, New York, N. Y.

10. Information obtained from the U. S. Environmental Protection Agency.

11. IFP, "Lube Hydrofinishing," Hydrocarbon Processing, September 1972, P. 167.

12. ASTM color after 96 hours.

Copy

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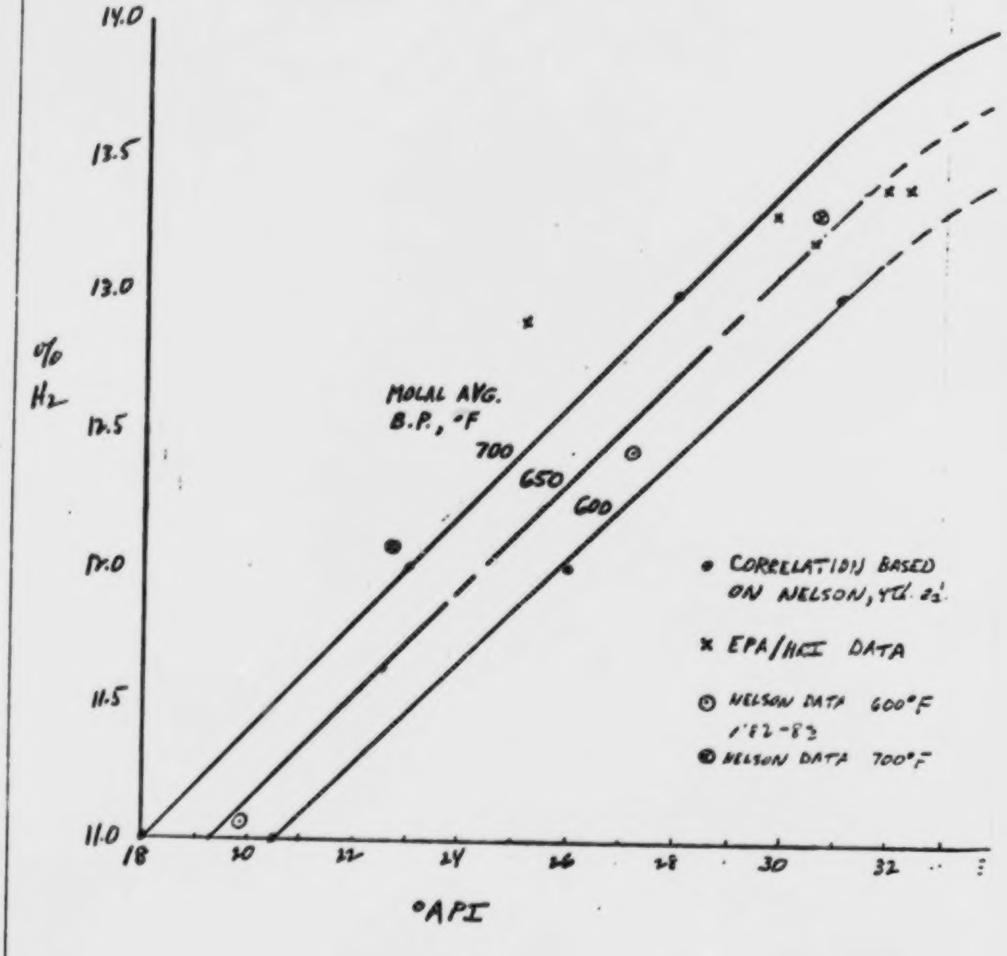
Princeton, N. J.
N. J. Weinstein
March 7, 1973

HYDROGEN CONTENT VS GRAVITY FOR LUBE OILS



HYDROGEN CONSUMPTION
BOMB TESTS

Hydrogen consumption bomb tests were conducted for NORCO by Merck & Co. Two tests were made; one control test with Nujol, and the other with a blend of NORCO lube distillates. The oil recovered from the NORCO oil hydrogenation was remarkably clear in appearance and odorless.

The interpretation of hydrogen consumption from bomb test pressures is very difficult because a number of simultaneous processes can occur, and apparently did occur in this work. These are catalyst reduction, hydrogen adsorption and chemisorption, H_2O formation and condensation, physical absorption of hydrogen into the oil, formation of NH_3 and H_2S (and possible absorption and adsorption), and saturation of aromatic and olefin compounds. As shown in Tables E-1 to E-5, and the attached figure, separation of these effects was attempted.

The maximum hydrogen consumption projected from the data for the NORCO blend was 70 to 160 SCF/B. It is believed that hydrogen consumption can be kept at a reasonable level as long as no aromatic oils are allowed to enter the lube distillate boiling range.

The principle uncertainty in the range of hydrogen consumption is lack of information on the extent of chemisorption. This and other uncertainties pertaining to adsorption, hydrogen solubility, and especially the effect of oil composition could be eliminated by a series of bomb tests. As can be seen from Table E-2, obtaining hydrogen consumption information from oil compositions is highly unreliable.

TABLE E-1

PROCEDURE FOR HYDROGEN

CONSUMPTION BOMB TEST

300 cc Bomb

1. Pressure test bomb with N_2.

2. Charge cold bomb with 50 grams oil and 50 grams of catalyst.

3. Charge bomb at room temperature (approximately 70°F.) with 303 psig. hydrogen. Record room temperature.

4. Heat bomb to 650°F. (343.3°C.) as quickly as possible without rocking. Record temperature and pressure as frequently as possible.

5. Start bomb rocking and hold temperature at 650°F. Record temperature and pressure as frequently as possible until pressure starts to line out. Keep system closed for at least 24 hours, longer if pressure has not lined out.

6. Allow bomb to cool to room temperature. Record pressure and temperature as frequently as possible.

7. Carefully release hydrogen pressure.

8. Recover oil and catalyst for analysis.

10. Run control with hydrogen and a paraffinic oil (Nujol,e.g.).

TABLE E-2

NORCO OIL HYDROGENATION TEST

OIL ANALYSIS

	BEFORE	AFTER
% C	86.30 ± 0.30	86.67 ± 0.30
% H	13.52 ± 0.30	13.67 ± 0.30
% N	0.03	<0.02
% S	0.27 ± 5%	0.036 ± 5%
% O	0 ± 0.2	0.2 ± 0.2

BOMB CHARGE

50 g. Harshaw No. HT-100 E 1/16" catalyst

50 g. Oil

Hydrogen (see Tables 4 and 5)

CATALYST ANALYSIS (Harshaw Brochure)

NiO, wt.%	3.8	Loss on Ignition @ 480°C., wt. %	1.4
MoO_3,wt.%	16.8	Charge Density, lbs./CF	38
SiO_2,wt.%	2.0	Surface Area, M^2/g.	191
Na_2O,wt.%	0.02	Pore Volume to 10,000 A°, cc/g.	0.54
Fe, wt.%	0.03	Average Pore Radius, A°	63

TABLE E-3

ANALYSIS OF HYDROGENATION

BOMB TESTS

			Assumed Mechanisms
Period I	Bomb filled with H_2 at room temperature to 320-320 psia.	1.	H_2 under pressure in vapor space.
		2.	H_2 adsorbed onto catalyst surface.
		3.	H_2 dissolved in oil.
Period II	Heatup of bomb	1.	Additional H_2 dissolves in oil.
		2.	H_2 desorption occurs.
		3.	Some hydrogenation.
Period III	Agitation at control temperature (616°K = 649°F.)	1.	Catalyst reduction ($H_2(V) + NiO \rightarrow H_2O(V) + Ni$)
		2.	Hydrogen chemisorption.
		3.	Hydrogenation.
Period IV	Cooldown of bomb	1.	H_2 adsorption.
		2.	H_2 dissolution.
		3.	H_2O condensation and adsorption.

TABLE F-4

RESULTS OF NUJOL HYDROGENATION TEST

		SCF/B
1.	H_2 vapor in bomb at end of Period I	= 634.0
	II	= 704.6
	III	= 615.1
	IV	= 460.1
2.	H_2 adsorbed and dissolved at end of Period I minus that at end of Period II = 704.6-634.0 (70.6 SCF/B = 1.2 x 10^{-3}lbs. H_2/lb. catalyst; 70.6 SCF/B = 18 mole % H_2 in oil; therefore, major effect is probably adsorption).	= 70.6
3.	H_2 chemisorbed = 704.6-615.1 (89.5 SCF/B = 3.0 atoms Ni/atom H - maximum usually considered to be 1.0).	= 89.5
4.	H_2 adsorption + H_2 dissolution + H_2O condensation and adsorption = 615.1-460.1	= 155.0
5.	H_2O condensation and adsorption = H_2O used in catalyst reduction = 155.0-70.6 (84.4 SCF/B = 1.43 moles H_2/mole NiO; however, some MoO_3 may occur also).	= 84.4

Assumptions:

1. n = PV/RT
2. No hydrogenation.
3. Chemisorption is irreversible under conditions used.
4. H_2 adsorbed and dissolved at end of Period IV essentially equal to that at end of Period I.
5. See Table 3.
6. Molecular weight of oil = 300.

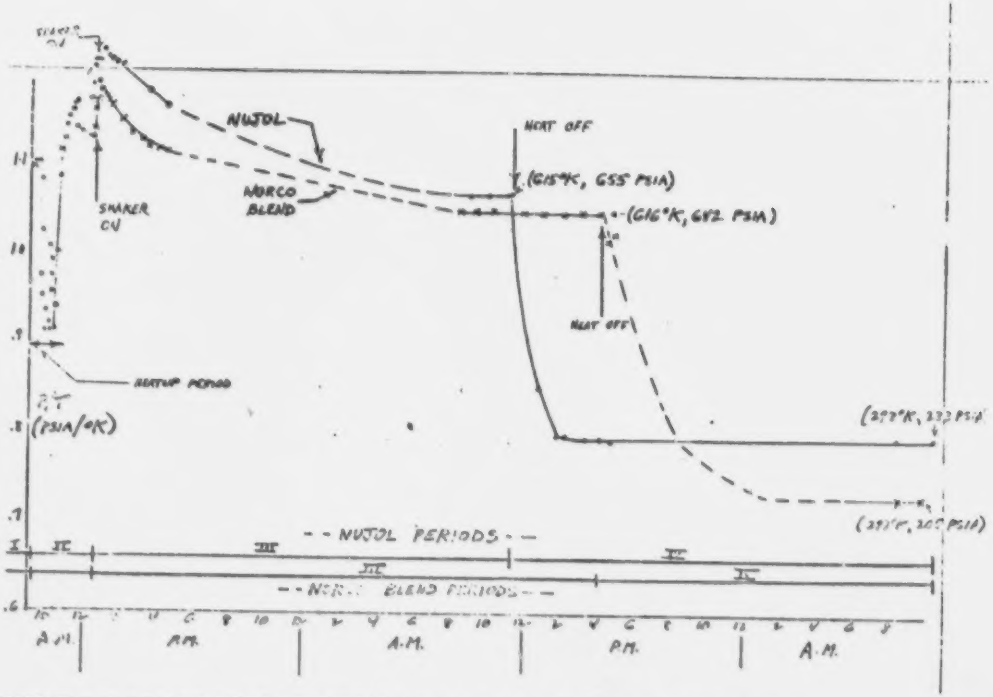
TABLE E-5

RESULTS OF NORCO OIL HYDROGENATION TEST

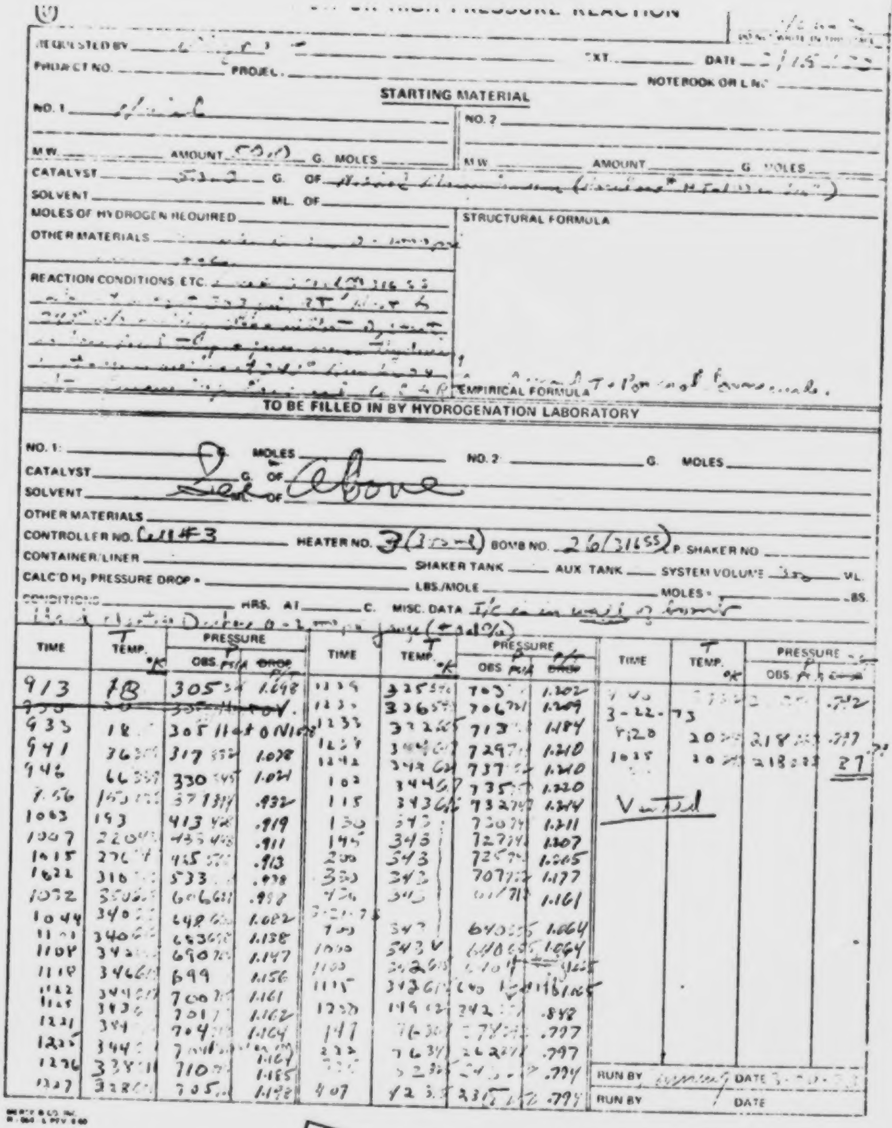
NORCO OIL: Blend of No. 3 (1.2857) and No. 4 (1.000)

		SCF/B
1.	H_2 vapor in bomb at end of Period I	= 630.0
	II	= 677.2
	III	= 600.6
	IV	= 397.0
2.	H_2 adsorption + H_2 dissolution + H_2O condensation and adsorption = 600.6−397.0	= 203.6
	Subtract H_2O used in catalyst reduction	= 84.4
		119.2
	Subtract H_2 adsorption + H_2 dissolution	= 70.6
	Then additional H_2O = H_2O from deoxygenation (48.6 SCF/B = 0.67% vs. 0 to 0.2% analyzed)	= 48.6
3.	Apparent H_2 consumption = 630.0−600.6	= 29.4
	Add H_2 available from H_2 desorption minus H_2 dissolved	= 70.6
	Add H_2O from deoxygenation	= 48.6
		148.6
	Subtract chemisorbed H_2	= 0−89.5
		= 59.1−148.6
	Add H_2 consumed in denitrogenation ($1.5\ H_2 \rightarrow NH_3$)	= 2.4
	Add H_2 consumed in desulfurization ($H_2 \rightarrow H_2S$)	= 8.5
	Predicted H_2 Consumption	70−160

Assumptions: See Tables 3 and 4.



HYDROGENATION BOMB TESTS



OF HIGH PRESSURE REACTION

REQUESTED BY [illegible] EXT. DATE [illegible]

PROJECT NO. PROJECT NOTEBOOK OR L.N.

STARTING MATERIAL

NO. 1 [illegible]

NO. 2

M.W. AMOUNT [illegible] G. MOLES

M.W. AMOUNT G. MOLES

CATALYST [illegible] G. OF [illegible]

SOLVENT ML. OF

MOLES OF HYDROGEN REQUIRED

STRUCTURAL FORMULA

OTHER MATERIALS [illegible]

REACTION CONDITIONS ETC. [illegible]

EMPIRICAL FORMULA [illegible]

TO BE FILLED IN BY HYDROGENATION LABORATORY

NO. 1: G. MOLES NO. 2 G. MOLES

CATALYST G. OF See Above

SOLVENT ML. OF

OTHER MATERIALS

CONTROLLER NO. Cell #3 HEATER NO. 3 ([illegible]) BOMB NO. 26 (31655) P. SHAKER NO.

CONTAINER/LINER SHAKER TANK AUX TANK SYSTEM VOLUME [illegible] ML.

CALC'D H_2 PRESSURE DROP = LBS./MOLE MOLES = LBS.

CONDITIONS HRS. AT C. MISC. DATA T/C [illegible] in wall of bomb

[illegible] gauge (± [illegible]%)

TIME	TEMP. T °K	PRESSURE OBS. P psia	PRESSURE ~~DROP~~ P/T	TIME	TEMP. T °K	PRESSURE OBS. P psia	PRESSURE ~~DROP~~ P/T	TIME	TEMP. T °K	PRESSURE OBS. P psia	PRESSURE P/T
913	18	305	1.698	1229	325	703	1.202	[illegible]	[illegible]	[illegible]	.792
~~[illegible]~~	~~[illegible]~~	~~[illegible]~~	~~[illegible]~~	1230	326	706	1.209	3-22-73			
933	18	305	[illegible]	1233	332	713	1.184	820	20	218	.797
941	36	317	1.078	1234	340	729	1.210	1035	20	218	.797
946	66	330	1.021	1242	342	737	1.210				
956	150	371	.932	102	344	735	1.220	Vented			
1003	193	413	.919	115	343	732	1.214				
1007	220	435	.911	130	343	730	1.211				
1015	270	485	.913	145	343	727	1.207				
1022	310	533	.978	200	343	725	1.205				
1032	350	606	.998	300	343	707	1.177				
1044	340	648	1.082	430	343	697	1.161				
1101	340	683	1.138	3-21-73							
1108	342	690	1.147	730	343	640	1.064				
1118	346	699	1.156	1000	343	640	1.064				
1142	344	700	1.161	1100	342	640	[illegible]				
1145	343	701	1.162	1115	343	640	1.065				
1221	344	704	1.164	1230	149	342	.848				
1225	344	7[illegible]	1.164	147	76	278	.797				
1236	338	710	1.185	233	76	262	.797				
1327	328	705	1.198	[illegible]	72	24[illegible]	.794				
				407	72	231	.794				

RUN BY [illegible] DATE [illegible]

RUN BY DATE

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HYDROGENATION OR HIGH PRESSURE REACTION

DO NOT WRITE IN THIS SPACE

REQUESTED BY [illegible] XT. DATE [illegible]/23/23
PROJECT NO. PROJECT NOTEBOOK OR L NO.

STARTING MATERIAL

NO. 1 [illegible] ([illegible])
M.W. AMOUNT [illegible] G. MOLES
NO. 2
M.W. AMOUNT G. MOLES
CATALYST [illegible] G. OF [illegible] ([illegible] # [illegible])
SOLVENT ML. OF
MOLES OF HYDROGEN REQUIRED
OTHER MATERIALS
REACTION CONDITIONS, ETC. [illegible]
[illegible] # [illegible]
STRUCTURAL FORMULA
EMPIRICAL FORMULA

TO BE FILLED IN BY HYDROGENATION LABORATORY

NO. 1: 50 G. MOLES NO. 2: G. MOLES
CATALYST 50 G. OF [illegible]
SOLVENT ML. OF
OTHER MATERIALS
CONTROLLER NO. # 3 HEATER NO. [illegible] BOMB NO. [illegible] L.P. SHAKER NO.
CONTAINER/LINER SHAKER TANK AUX. TANK SYSTEM VOLUME [illegible] ML.
CALC'D H_2 PRESSURE DROP = LBS./MOLE MOLES = LBS.
CONDITIONS HRS. AT [illegible] C. MISC. DATA [illegible]

TIME	T TEMP. °K	PRESSURE OBS. P PSIA	PRESSURE P/T ~~DROP~~	TIME	T TEMP. °K	PRESSURE OBS. P PSIA	PRESSURE P/T ~~DROP~~	TIME	T TEMP. °K	PRESSURE OBS. P PSIA	PRESSURE P/T ~~DROP~~
[illegible]	[illegible]	[illegible]		100	343 66	706 12	1.172	4:45	24[illegible]	110	.690
7:23	21 [illegible]	306 341	1.095	110	344 [illegible]	706 731	1.171	Vented			116
7:38	107 [illegible]	354 369	.972	120	34[illegible]	7[illegible] 717	1.164				
9:41	151 [illegible]	565 [illegible]	.947	130	343	[illegible] 714	1.161				
9:54	224	437 452	.911	200	343	657 704	1.145				
1008	227	492 567	1.005	230	343	681 696	1.130				
1010	32[illegible]	550 565	.954	305	343	675 690	1.123				
1015	340 61[illegible]	552 597	.972	330	343	672 687	1.117				
1020	352 [illegible]	603 618	.998	400	343	670 685	1.114				
1045	342	667 682	1.110	430	343	668 683	1.112				
1050	344 [illegible]	[illegible]	1.122	5-27-7							
1129	344	625 [illegible]	1.137	[illegible]	343	631 646	1.048				
1150	344	683 6[illegible]	1.143	410	343	631 646	1.048				
1217	344	678 693	1.127	1015	343	631 646	1.048				
1228	348	677 [illegible]	1.126	1155	343	629 644	1.046				
1229	340	643 [illegible]	1.136	100	343	628 643	1.045				
1230	328	678 6[illegible]	1.155	210	343	627 [illegible]	1.044				
1231	326	677 [illegible]	1.161	310	343	627 [illegible]	1.044				
1233	328	679 [illegible]	1.163	410	343	[illegible] 764	1.044				
1240	344	702 717	1.162	437	270 [illegible]	538 553	1.017				
1241	342	701 726	1.164	452	244 51	4[illegible] 50[illegible]	1.022				
1251	342	707 722	1.177	[illegible]	24[illegible]	110 20	.690				

RUN BY [illegible] DATE [illegible]
RUN BY DATE

MERCK & CO. INC.
R-854-L REV. 6-80

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Mr. Solfred Maizus, Project Director
National Oil Recovery Corporation
P. O. Box 338
Bayonne, New Jersey

Re: FWQA #15080 DBO
Diesel Engine Fuel Tests
Of Refinery Products #3 and #4

Dear Sir:

As our company is desirous of participating in your testing program to demonstrate that your special fuels which are produced from crankcase waste oils- can be used as a diesel engine fuel, the following is a report based on day-to-day usage of your #3 and #4 refinery products - used in mixture with regular diesel fuel:

Test Report on Various Fuels Used for Diesel Fuel Truck H-5 1958 Diamond T 1962 Cummings C-180 Diesel Engine

5/18/70	Emptied fuel oil tank and put in 50-50 mixture totalling 40 gallons of regular diesel and LDA #3.
5/19/70	Put in same mixture totalling 20 gallons, mileage 135,520.
5/20/70	Put in same mixture total 24 gallons, mileage 135,640.
5/21/70	Put in same mixture total 18 gallons, mileage 135,730.
5/22/70	Put in same mixture total 35 gallons, mileage 135,923.
5/23/70	Disassembled fuel lines off and checked for

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5/23/70	varnish, removed fuel filter and took apart. No varnish, no foreign matter on steel.
5/25/70	Based on a mixture of 75% LDA #3 and 25% of regular diesel, 24 gallons was put in of the mixture specified, mileage 135,923.
5/26/70	Put in same mixture totalling 20 gallons, mileage 136,043.
5/27/70	Put in same mixture totalling 18 gallons, mileage 136,151.
5/28/70	Put in same mixture totalling 19 gallons, mileage 136,265.
5/29/70	Put in same mixture totalling 22 gallons, mileage 136,397.
5/30/70	Removed fuel filter and check out, all parts are clean, no varnish. Seems to be getting 10% more mileage out of same gallon of fuel. Drivers have advised there also seems to be more power. Complaints are as follows: The mixture has a bad odor coming out of the exhaust pipe, when running and idling, very offensive. It is also showing traces of black smoke being emitted from stack of exhaust pipe.
6/1/70	Based on a mixture of 100% LDA #3. 23 gallons, mileage 136,540.
6/2/70	36 gallons, mileage 136,765.
6/3/70	30 gallons, mileage 136,952.
6/4/70	25 gallons, mileage 137,108.
6/5/70	20 gallons, mileage 137,228.

> The smoking out of the exhaust stack seemed to turn to slightly white, but the odor was very objectionable. The power as claimed by the driver seemed to be very good in pulling hills. Fuel filter was disassembled and seemed to be clean. A carbon smell prevailed.

We will continue to test your productr on our diesel trucks until instructed otherwise. Let us again re-state our willingness to contribute supplies and services to your worthwhile program - which demonstrates the recycling of a wasted natural resource into useful saleable products.

Table G-1. CHARACTERIZATION OF WASTE FUEL OIL IN 20 X 20 SOUTH TANK (Charge for Run No. 9)

Tank: 20 ft. diameter x 20 ft. high

SAMPLE	LOCATION	API GRAVITY	% WATER*	% OIL*
1	Top	29.5	-	100
2	18 ft. from top	26.0	10	90
3	18 ft. from top	26.4	10	90
4	17 ft. from top	25.1	10-15	87-95
5	12½ in. from bottom	8.5	-	100

* By centrifugation at 32,000 G for 30 minutes.

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Table G-2. CENTRIFUGATION OF A WASTE OIL SAMPLE (Northern New Jersey Oil Co.)

From Tank Top	From Tank Middle	From Tank Bottom	Centrifuge Results
22.5 ml.	20	11-13	Liquid Oil
13.0 ml.	11.5-12	5-7	Heavy oil or wax
1. ml.	2	12	Water

Table G-3. AROMATIC DISTILLATION BOTTOMS WASTE OIL

Specific Gravity @ 60°F	1.136
API Gravity, °F	<0
Flash, COC, °F	420
Pour Point, °F	+55
Sulfur, %	0.4
Saybolt Furol Viscosity @ 122°F	130
B.T.U./lb.	17,000
Distillation	
Initial, °F	630
5%	642
50%	674

Not miscible in NORCO #3 sidestream

Table G-4. COAL TAR OIL

Hydrogen, wt. %	6.24	
Nitrogen	0.18	
Sulfur	0.36	
Carbon	85.9	
Ash	0.028	
Distillation (100 ml)		
initial, °C	50-59	
3 ml. overhead	150	(slight yellow color)
97 ml. liquid	-	(dark color)

Table G-5. WASTE OIL INSPECTIONS

Sundry samples from suppliers and NORCO tanks were analyzed as follows:

Sample Identification	
S - 20 x 20 Tank	29.5 API gravity 100% oil
S - 20 x 20 - 18'	26.0 " " 90% oil, 10% H_2O
S - 20 x 20 - 18'	26.4 " " 90% oil, 10% H_2O
S - 20 x 20 - 17'	25.1 " " 85% oil, 10% H_2O
Bottoms of S 20 x 20	8.5 " " 100% oil
16 x 16 Tank	6.84% Ash
J. Noonan B Tank	20% H_2O, 0.215% Ash
#6 Oil Sample (NBR-86-2)	100% oil, 0.005% Ash
J. Trainor (NBR-94-1)	85% oil
Howard Fuel Corp. (loan 41) (NBR-94-2)	No water
J. Trainor #3	Trace H_2O
R & H	Trace H_2O
J. Trainor (NBR-94-5)	Trace H_2O
Solar Chemical (Tank 209)	60% oil, 10% sediment, 30% water
B. Ogust	85% oil, 5% sediment, 10% water, 0.127% Ash
Ryan (Citgo)	1.1% BS&W
Howard Fuel (loan 14)(NBR-99)	50.2% BS&W
Trainor, Jr.	15.0% BS&W
Trainor, Sr.	3.0% BS&W
Smerdon	4.0% BS&W
Lenza	6.0% BS&W
Smerdon	5.0% BS&W
Trainor, Jr.	5.0% BS&W
Admiral	20.0% H_2O, Gravity 21.6
National - 1	2.1% BS&W
National - 2	54% BS&W
Oil Tank Cleaning	33.8% H_2O, 0.90% Sediment
Industrial Separator	1.4% H_2O, 0.74% Sediment

Table G-6. WATER CONTENT OF OILS RECOVERED FROM BARGE CLEANING

VALVE 1	90% H_2O
VALVE 2	56.6% H_2O
VALVE 3	0 % H_2O
VALVE 4	0 % H_2O

Table G-7. WASTE OFF-SPEC PETROLEUM PRODUCTS

An inspection and sampling trip was made to the Citgo terminal at Linden, N.J. where 13 samples were taken. Eight tanks were gauged and average or top middle and bottoms samples were taken. Notes were made on existing terminal pumps and lines. Data was accumulated on leading hoses and equipment required to economically load tank trucks with oil from the subject tanks.

The Citgo terminal was formerly the site of a relatively large asphalt production and also a terminal operation. The asphalt operation and associated tankage has been eliminated. The terminal operation with dock and tanks continues. Eight scattered tanks contained varying quantities of mixed and contaminated oils which do not meet standard product specifications covering: flash, gravity, distillation range, etc. The scattered tanks and limited access pose transportation problems. Truck filling from the various tanks will require from 50 to 300 feet of loading hose with fittings depending on location and pump pressures available.

Table G-8. WASTE TAR OIL FROM AN OBSOLETE COAL GAS PLANT

The gas holder at the Long Branch, New Jersey plant was of the conventional liquid sealed telescoping type. The bottom section, about 147' - 150' in diameter by 35' high, was almost completely filled with liquid, as is the case during normal holder operation. It contained about 24' of water and 10'7" of dark viscous sticky fluid slightly heavier than water. This dark fluid settled to the bottom of the bottom section. There seemed to be about 6" semi-solid ooze on the bottom. The line of demarcation between relatively clear water and dark sticky material was not sharp. Even at 1' above the floor of the bottom section, turbid water interpenetrated globs of the dark sticky viscous material.

Samples of dark viscous fluid taken about 16" above the bottom ooze contained pockets of turbid water. The water tasted almost like rain water, felt the same, and on smooth plane surfaces spread out and behaved like tap water. It had little odor. Other material from a small tank being dismantled had the same characteristics.

The plant was provided with what looked like a shell still with condensing coil and fractionating tower.

From the absence of volatile material, in samples taken, it was concluded that lower boiling aromatic materials were promptly removed from coal tar and disposed of when the plant was operating.

Table G-9. CHEMICAL PLANT WASTE OILS -- ANALYZED AT POINT OF GENERATION

Nature & No	ELEMENTAL ANALYSIS (%)										Heating Value	Viscosity
	C	H	O	N	S	Sulfated ASH	Alkali Metals			Chlorine	BTU/LB	(cp. °C)
							Na	K	Ca	Cl		
Solvent Wastes												
1	65.92	15.50	16.15	-	0.41	0.9	-	-	-	2.04	14,650	4.2^{22}
2	89.46	9.18	1.87	-	0.42	-	-	-	-	0.33	17,000	2.5^{23}
3	36.70	12.20	47.0	3.37	0.23	-	-	-	-	0.13	10,250	9.0^{23}
4	37.24	6.94	37.11	8.86	0.38	14.14	-	3.5	-	1.51	8,100	40.0^{23}
5	21.15	10.07	22.50	0.68	0.96	7.69	1.47	-	-	36.8	4,570	20^{23}
Heavy Residues												
6	81.50	5.10	8.76	-	7.94	0.46	-	-	-	-	13,500	270^{100}
7	68.87	6.55	15.16	-	5.03	13.78	2.66	-	-	-	13,125	10000^{105}
8	80.93	10.57	8.90	-	0.53	2.4	0.46	-	-	-	15,650	(700 [illegible]
9	67.37	3.82	15.22	6.70	0.23	2.69	0.47	-	-	0.77	11,810	625^{23}
10	71.18	9.23	15.16	0.21	2.50	11.65	2.24	-	-	0.75	13,280	4400^{165}
11	79.87	5.48	8.76	9.07	0.37	0.19	-	-	-	-	13,500	50^{23}
12*	55.5	4.75	7.65	5.30	-	14.10	-	-	-	15.72	10,740	110^{105}
13	62.02	4.18	1.20	10.55	19.74	2.43	0.44	-	-	-	11,770	150^{23}
14	70.56	8.52	9.32	7.85	-	0.78	-	-	-	0.15	15,480	1100^{100}
15	36.16	2.00	2.20	5.81	53.71	-	-	-	-	-	8,400	$300\text{-}500^{90}$

* Contains 5.7% Zn

Table G-10.CHEMICAL PLANT WASTE OILS

Identification	BN Pitch No. 1	BN Pitch No. 2	PEP Residue No. 3	SBS Tars No. 4
Wt. % Sulfur	0.002	-	-	3.57
Wt. % Ash	4.3	4.4	12.3	0.4
Semi-Quantitative Spectographic Analysis of Ash, wt. %				
Principal 10-100	-	-	Na	
Major 1-10	Fe,Na	Na	Al,Si	
Strong 0.1-1.0	Al,Si,Cu	Fe,Cu	Fe,Ca	
Medium 0.01-0.1	Ni,Mg,Pb, Mn,Ti,Ca	Al,Ni,Si, Mn,Ca	Ni,Cr,Mg,Pb, Mn,Cu,Ti	
Weak 0.001-0.ol	Cu,Cd,B,P, Mo,Ba	Cd,Mg,Pb, Ti	Cd,Sn,B,Mo, V,Ba	
Trace 0.0001-0.001	In,V,Zr,Co	B,Mo	Ag,Zr,Co	
Faint Trace <0.0001	Ag	Ag	-	
Not Detected	Bi,Ge,Ga, Sn,As,Sb,W, Au,Hg,Be, Zn,K	Bi,Pt,Ge, In,Cr,Sn, As,Sb,Ga, W,P,Au,Hg, Be,V,Zn,Zr, Co,K,Ba	Bi,Ge,In,Ga, As,Sb,W,P,Au, Hg,Be,Zn,K	
Miscibility*				
Varsol	Very Slight	-	-	None
Petroleum Ether	None	-	-	None
Toluol	Slight	-	-	Slight
Benzene	Fair	-	-	Good-Excellent+
MEK	Excellent	-	-	Good-Excellent+
Xylene	Poor	-	-	Fair
MEK/NORCO #6	2 ml of (1 gram Pitch+ 3ml MEK) + 3 ml NORCO #6-Good Mix	-	--	2 ml of (1 gram Tar + 3 ml MEK) + 3 ml NORCO #6-Good Mix

* Pitch and tars ground in mortar and pestal prior to miscibility tests.
+ Some precipitate noted.

Table G-11. IODINE NUMBER OF NORCO CRANKCASE OILS

	IODINE NUMBER, g./100g. oil
Centrifuged Oil	0.73
NORCO No. 3	0.91
NORCO No. 4	1.03

APPENDIX H

COALESCING PLATE OIL/WATER SEPARATOR

The attached figures illustrate the separator design. It consists of three principle sections--an influent region, the coalescing section itself, and an effluent region. Each is described below.

The oil/water mixture enters the separator through an "H" shaped inlet manifold. A series of holes drilled in the vertical members of this manifold (a) helps distribute the water uniformly over the entire water cross-section of the tank (b) degasses the influent and (c) dampens pulsating flows. A vertical slot flow straightener located immediately downstream of the inlet manifold completes flow distribution and insures uniform conditions into the coalescing section.

The coalescing region begins with the first group of corrugated plates. Eight individual plate stacks, arranged in two rows of four each, are used in the OPC-50. Each stack consists of a series of plates stacked vertically, three plates to the inch, with the convolutions running horizontally at right angles to the flow. Spacing between the plates is maintained by tabs molded into each plate. The entire stack assembly is held together with two tie rods running between a tubular lower support and a channel at the top. Stack dimensions are 1 foot by 2 feet by 5 feet high. The dry weight of each stack is approximately 100 pounds. Short stainless steel shims are placed between plate stacks to lock the entire assembly together.

As the oil/water mixture passes through the plates, oil particles coalesce and tend to collect at the crest of each corrugation. Bleed holes are provided along the crests so that, as the water passes through the plates, the oil rises progressively upward through the holes in the plates to collect and form an oil blanket on the water surface.

Two plastic foam coalescing packs can be installed after the corrugated plate section described above. These packs are designed to improve the coalescence of finely emulsified oil droplets or oil coated particles. Both packs are identical, consisting of a foam pad mounted in a split frame. This arrangement allows for easy removal of the foam for cleaning or replacement. In addition, the foam is extended beyond the frame to provide a seal with the tank walls, preventing any water bypass flow.

A second corrugated plate coalescing section consisting of one row of four plate stacks is located downstream of the packs to complete the separating action. The individual stacks are identical to those previously described.

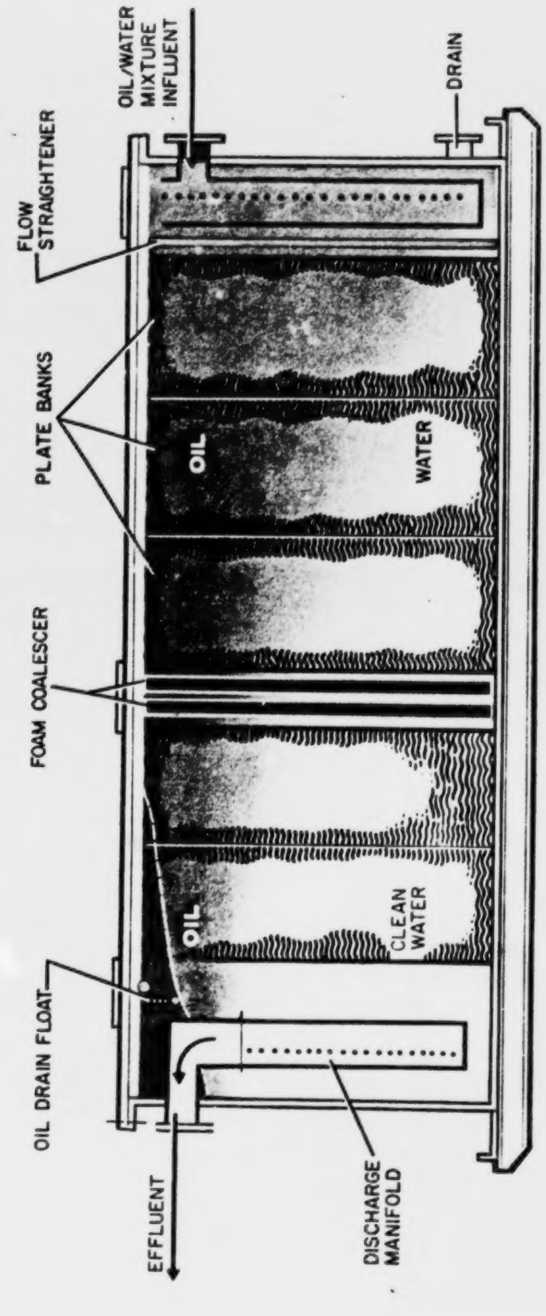
The water outlet is an inverted "U" shape pipe with a series of holes to admit the water without disturbing the oil film. The holes are located so that the upper portion of the manifold acts as an oil dam, preventing the oil from mixing with the effluent water.

Oil is removed from the unit by a float-type oil skimmer. Two ball type floats are used to balance the unit. The oil flows through a slotted tube between these floats, then through a flexible hose to the discharge fitting. The vertical position of the hose on the slotted tube may be changed to vary the length of the slot and hence the thickness of the oil film. With this exception, operation of the skimmer is completely automatic.

The entire unit is fitted with a gasketed cover and may be operated with a low pressure gas blanket. Access ports for removal of the coalescing pack and access to the skimmer are included in the cover.

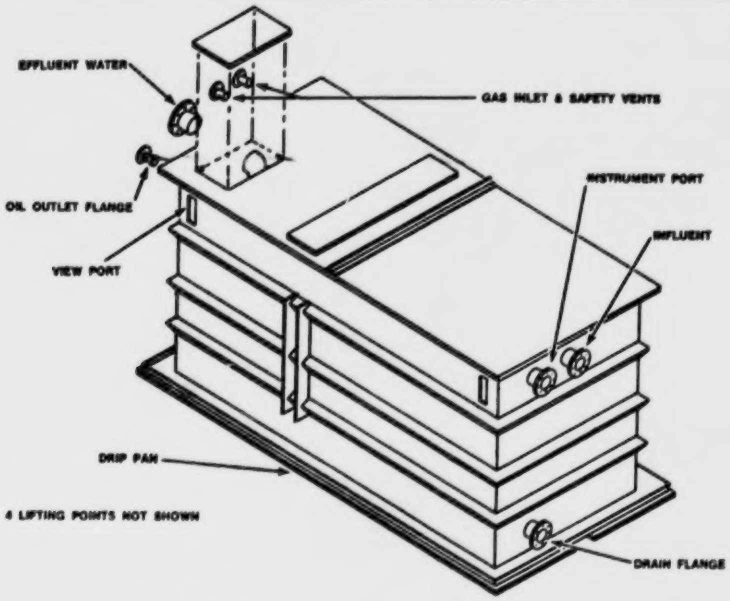
The unit is contained within a rectangular, skid mounted tank. A continuous drip pan is located beneath the tank. Overall dimensions of the unit are 127" long by 62" wide by 68" high. The dry weight of the assembly is approximately 4700 pounds.

The tank is fitted with all necessary flange connections. These flanges are fiberglass, with a standard ASA 150 pound flange bolt circle configuration. Location, size, and intended purpose for each flange is shown on the figures. The intended purpose is self-explanatory with the exception of the instrument port flange, which is used primarily for a high level shutdown if installed. The size and location of the removable cover sections and the view ports are also shown. Note clearance requirements for coalescing pack removal. Four lifting lugs are provided on the skid for use in handling the unit.


OIL/WATER MIXTURE INFLUENT
DRAIN
FLOW STRAIGHTENER
PLATE BANKS
OIL
WATER
FOAM COALESCER
OIL
CLEAN WATER
OIL DRAIN FLOAT
EFFLUENT
DISCHARGE MANIFOLD

Dimensions

MODEL	APPROX. DRY WEIGHT (lbs.)	LENGTH (inches)	HEIGHT (inches)	WIDTH (inches)	INFLUENT FLANGE (IPS)	EFFLUENT FLANGE (IPS)
OPC-10	850	68	44	32	2"	4"
OPC-30	1350	90	56	48	4"	6"
OPC-50	4700	127	68	63	4"	6"
OPC-100	8500	180	70	72	6"	8"


EFFLUENT WATER
GAS INLET & SAFETY VENTS
OIL OUTLET FLANGE
INSTRUMENT PORT
VIEW PORT
INFLUENT
DRIP PAN
DRAIN FLANGE

4 LIFTING POINTS NOT SHOWN

Coalescent Plates Increase Capacity of Tanks or Pits

The unique coalescent plates which are the heart of the General Electric Oil/Water Separation system can be added to any existing gravity settling tank or pit to considerably increase capacity without modification or enlargement of the existing system.

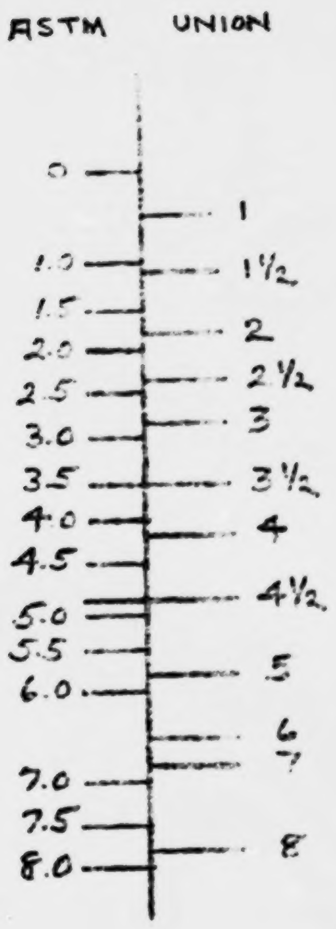
The coalescent plates are available in any size banks or modules, each individual plate being 1 ft. x 2 ft. These can be stacked in virtually any configuration to permit flexibility of installation and to accommodate the desired flow rate.

Gravity separation is increased by the use of these plates, without the addition of chemicals. Fabricated with non-corrosive materials and with no moving parts or filters, operation is virtually maintenance free.

Cost is $80.00 per foot of height of the stacked coalescent plates.

APPENDIX I

ASTM VS. UNION COLOR CORRELATION


ASTM
UNION
0
1
1.0
1½
1.5
2
2.0
2½
2.5
3
3.0
3.5
3½
4.0
4
4.5
4½
5.0
5.5
5
6.0
6
7
7.0
7.5
8
8.0

APPENDIX J

PROCESS SCREENING STUDIES

REPORT TO NORCO ON THE POTENTIAL PROFITABILITY OF SEVERAL APPROACHES TO RE-REFINING CRANKCASE WASTE OIL

FOR: NATIONAL OIL RECOVERY CORPORATION
P. O. BOX 338
BAYONNE, NEW JERSEY 07002

Date : January 22, 1973

Work By : Dr. N. J. Weinstein
Dr. A. Boyum

Report By: Dr. N. J. Weinstein
RECON SYSTEMS, INC.
Cherry Valley Road
Princeton, N. J. 08540

REPORT TO NORCO ON THE POTENTIAL PROFITABILITY OF SEVERAL APPROACHES TO RE-REFINING CRANKCASE WASTE OIL

Summary

Based on NORCO's projections of 3¢/gal. or less to be paid for crankcase waste oil, and 16¢/gal. or more realization for recovered lubricating oils, re-refining is potentially very profitable. For example:

Pre-treatment	-----None-----			Solvent
Post-treatment	-----Hydrogen-----			Hydrogen
Purchase of Exxon Equipment	Yes	Yes	No	Yes
Feed, gals/yr.	9,000,000	29,000,000	29,000,000	34,800,000
Total Investment, $	492,000	1,123,000	3,050,000	1,773,000
Profit, $/yr.	129,000	1,573,000	1,488,000	2,194,000
Return, %/yr.B.T.	26	140	49	124

The most critical factors to be resolved before such profitability can be realized are:

- -assuring a supply of crankcase waste oil, preferably in excess of 20 million gallons per year at a cost of 3¢/gal. or less.
- -assuring a comparable market for re-refined lubricating oils at 16¢/gal. or more.
- -the development of technology which will convert the crankcase waste oils to lubricating oils meeting specifications necessary to command the above price.

As to the question of technology, we feel that vacuum distillation followed by hydrogen treating has a high probability of early success. The availability of most of the necessary equipment from Exxon's Bayonne Refinery makes this possibility particularly attractive, if sufficient land area can be assembled. Chemical treatment is also promising as an independent method of re-refining, or as an adjunct to vacuum distillation plus hydrogen treating, but this approach will require a more extensive development program.

The development of vacuum distillation plus hydrogen treating technology requires solutions to two major problems: (1) assured operability of the heating and distillation equipment to avoid premature shutdowns due to coking and fouling; and (2) upgrading lubricating oil cuts to meet color, odor, and other specifications.

We feel that the operability problem can be alleviated sufficiently by furnace redesign and the use of anti-fouling additives to obtain the modest 5000 hour per year operation assumed in the economic study. We recommend an engineering design study to determine the furnace parameters required for future operations.

Hydrogen treating is widely used for upgrading virgin lubricating oil and petroleum fuels to meet odor, color, nitrogen, sulfur, stability and other important specifications. The brief experimental hydrogen testing program which NORCO sponsored in April 1972 supports the possibility that lubricating oil cuts from crankcase waste oil distillation can be similarly upgraded. Further experimental work is recommended prior to a commitment for purchase of Exxon's equipment and erection at a site assembled by NORCO.

Description of Alternatives

Four basic alternatives, shown diagrammatically in Figures 1 and 2, were studied as possible approaches to re-refining crankcase waste oil. These are:

CASE A - BASE CASE

Essentially present operation at full capability of 1030 B/SD for 5000 hrs/yr. using vacuum distillation (including vacuum flashing) alone. This operation produces lubricating oil with unsatisfactory color.

CASE B.1.1.

Envisions purchase of Exxon's Monophiner (catalytic hydrogen treating unit) to upgrade lube oil quality. Proposed operation shown in Figure 3. Existing vacuum distillation equipment operates 5000 hrs./yr.; hydrogen treating is oversized and required only 1550 hrs./yr. operation. Anti-foulant is used to ease operability problem on vacuum distillation equipment, but frequent shutdowns would still be required for cleaning.

CASE B.1.2.

Envisions purchase of both Exxon's Monophiner and vacuum distillation equipment. Lube oil is run 1550 hrs./yr. on both vacuum distillation equipment and hydrotreating. Excess capacity on vacuum distillation equipment is used to upgrade fuel oil by drying (3450 hrs./yr. at 3322 B/SD). Operability problems with lube oil on vacuum distillation equipment is expected to be eased by both anti-foulant and improved furnace design, but the extent of improvement requires further engineering design and/or experimental verification.

CASE B.2.2.

Envisions purchase of both Exxon's Monophiner and vacuum distillation equipment to be operated at full capacity, limited by heat exchanger and furnace considerations (3322 B/SD for 5000 hrs./yr.; 2115 B/SD limitation calculated for hydrogen treating at 700°F). Operatility limitations as in Case B.1.2.

CASE C.1.

1030 B/SD crankcase waste oil is treated at ambient conditions with n-butanol at a volumetric ratio of 2/1, as shown in Figure 4. The n-butanol is recovered by distillation and recycled. A semi-dry solid product high in lead is recovered from the oil by centrifugation. Lube oil is recovered from the treated crankcase oil by existing vacuum distillation equipment. Operability, product quality, and solids recovery are questionable, requiring further development work. However, 6000 hrs./yr. operation was assumed for this case, as well as improved lube oil yield (71.8% vs 63.9%).

CASE C.2.

Envisions purchase of Exxon's vacuum distillation equipment to increase Case C.1. operation from 1030 to 3322 B/SD for 6000 hrs./yr.

CASE C.'1.

Same as Case C.1. except that Exxon's Monophiner would be purchased to upgrade lube oil quality (2120 hrs./yr.).

CASE C.'2.

Same as Case C.2. except that Exxon's Monophiner would be purchased to upgrade lube oil quality (6750 hrs./yr.).

CASE D.

Same as Case B.2. except that all equipment is purchased new on a grass roots basis, including investment for offices and laboratory.

In the above cases where refinery expansion beyond the present 1030 B/SD was not contemplated, no pollution control facilities were added, except for a scrubber on the hydrogen treating purge gas. Where expansion to 3322 B/SD was contemplated an air flotation unit was added to handle oil-water separation problems.

Results

A summary of the case descriptions and the required investments are shown in Table 1. The investments for removing and relocating Exxon equipment were estimated to be about 53% of the required investment for new equipment, excluding new equipment to be purchased. All investments were based on indices for the 4th quarter of 1972.

The potential profitability of each case studied is shown in Table 2. The calculated return for all cases was attractive, but returns were especially attractive for larger operations. Some direct comparisons which can be made are:

	Case	Profit Before Tax ¢/gal. feed	Profit Before Tax $/yr.	Return, %/yr. before Tax
Purchase of Exxon equipment	B.2.	5.43	1,573,000	140
Grass roots plant	D.	5.13	1,488,000	49
9,000,000 gal/yr. feed	B.1.1.	1.44	129,000	26
29,000,000 gal/yr. feed	B.2.	5.43	1,573,000	140
No pre-treat.	B.2.	5.43	1,573,000	140
Solvent pre-treat. to improve yields and operability	C'.2.	6.30	2,194,000	124

A sensitivity analysis was carried out on Case C'.2., since this operation contained most of the major elements studied. The results of this analysis are shown in Table 3.

Doubling process investment, decreasing yields, doubling operating labor costs, doubling indirect costs, increasing feed cost from 3 to 5¢/gal., increasing hydrogen consumption by a factor of ten, and increasing solvent lost by a factor of five, all had significant effects on profitability, but none of these changed the basic attractiveness of the operation. On the other hand increasing lube oil realization from 16¢ to 20¢/gal. increased potential before tax return from 124 to 191%/yr.

Cost bases and cost details are provided in Tables 4, 5, and 6.

Working capital and land costs were not included in this analysis. Plots required are approximately 90'x120' for Exxon's Monophiner, 100'x160' for the vacuum distillation unit, and 30'x50'for hydrogen storage. The total land required with spacing, but excluding storage tanks, is about 40,000 to 50,000 square feet.

Conclusions

1. There appear to be strong economic incentives for refining crankcase oil to produce saleable lube oils.

2. A spread of 13¢/G between crankcase oil and lube oil easily justifies re-refining in a 29MMGAL/yr. (1900 B/CD) refinery.

3. Investments and operating costs projected for vacuum distillation, hydrofining, and solvent treating, or combinations of these processes, can easily be justified if they could produce 60% or greater yields of lube oil (13¢/G spread).

4. Insufficient data is available to be assured that any of the schemes studied can produce these saleable lube oils.

5. The prognosis for technical and economic success of R + D in this field is highly favorable.

6. The availability of monophining and vacuum distillation equipment from Exxon's Bayonne Refinery may provide NORCO with an unusually favorable position in this field, if technical feasibility can be quickly demonstrated in the laboratory.

Recommendations

1. NORCO should immediately undertake or preferably sponsor hydrofining experimentation to demonstrate the basic feasibility of this approach. Data is needed on H_2 consumption, catalyst life, bed fouling, reactor conditions, etc.

2. At the same time, NORCO should exhaustively examine the financial and engineering feasibility of adapting Exxon's equipment (monophiner + vacuum distillation), and of obtaining sufficient assured supplies of crankcase oil and other materials to provide about 25MMgal/yr. or more refined products.

3. If (1) and (2) above are favorable, NORCO should proceed with this project.

4. NORCO and EPA should formulate a long term R + D program designed to provide the data needed to assure the technical success of modern approaches to re-refining.

5. Vacuum distillation and hydrofining may be coupled with chemical methods to provide reasonable lube oil yields.

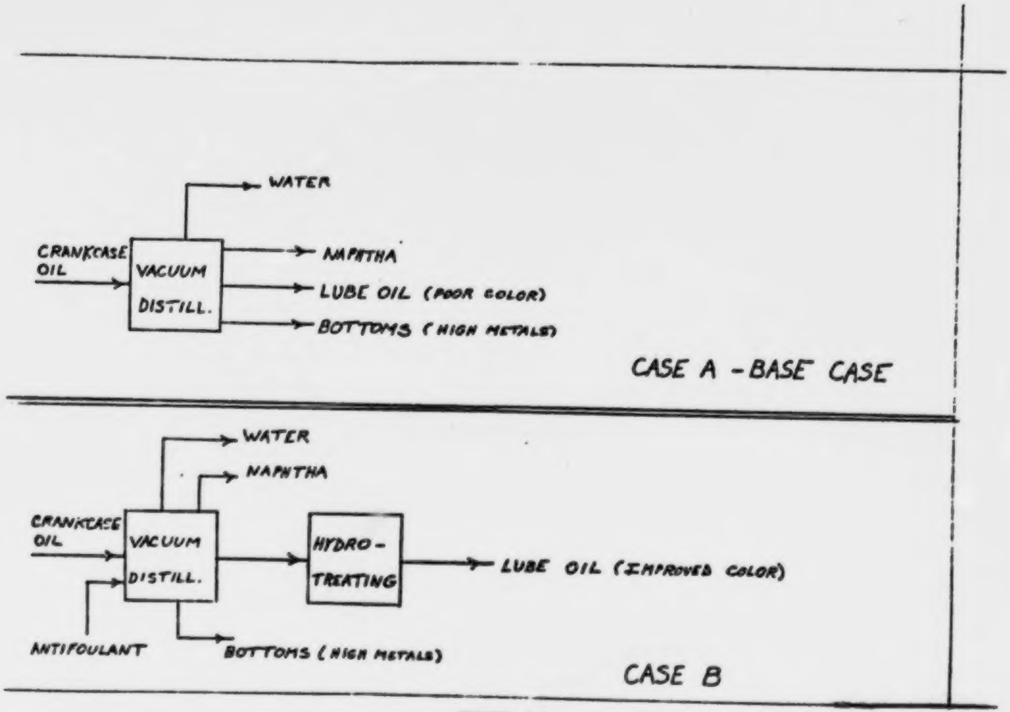

WATER
CRANKCASE OIL
VACUUM DISTILL.
NAPHTHA
LUBE OIL (POOR COLOR)
BOTTOMS (HIGH METALS)
CASE A - BASE CASE
WATER
NAPHTHA
CRANKCASE OIL
VACUUM DISTILL.
HYDRO-TREATING
LUBE OIL (IMPROVED COLOR)
ANTIFOULANT
BOTTOMS (HIGH METALS)
CASE B

FIGURE J-1

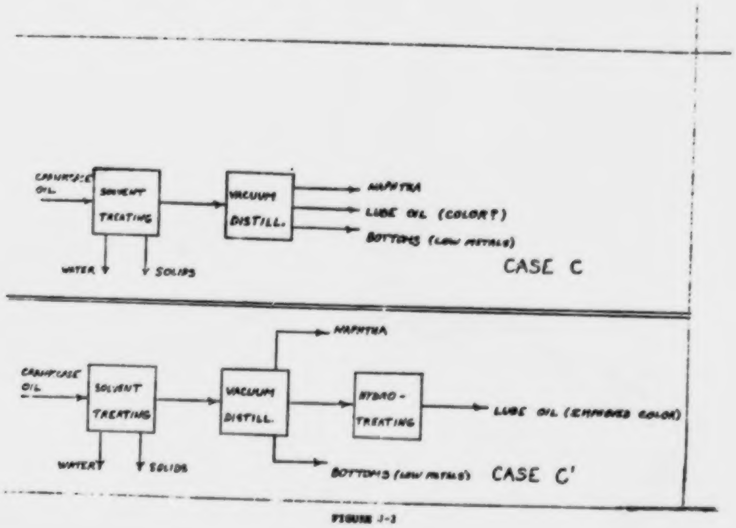

CRANKCASE OIL
SOLVENT TREATING
VACUUM DISTILL.
NAPHTHA
LUBE OIL (COLOR?)
BOTTOMS (LOW METALS)
WATER
SOLIDS
CASE C
NAPHTHA
CRANKCASE OIL
SOLVENT TREATING
VACUUM DISTILL.
HYDRO-TREATING
LUBE OIL (IMPROVED COLOR)
WATER
SOLIDS
BOTTOMS (LOW METALS)
CASE C'

FIGURE J-2

CASES C.1 AND C.1. - SOLVENT TREATING

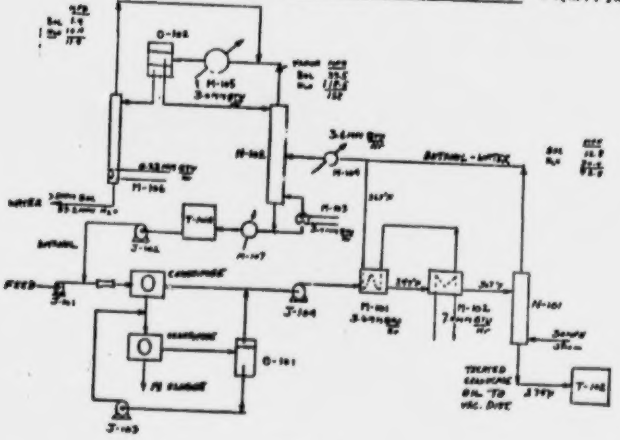
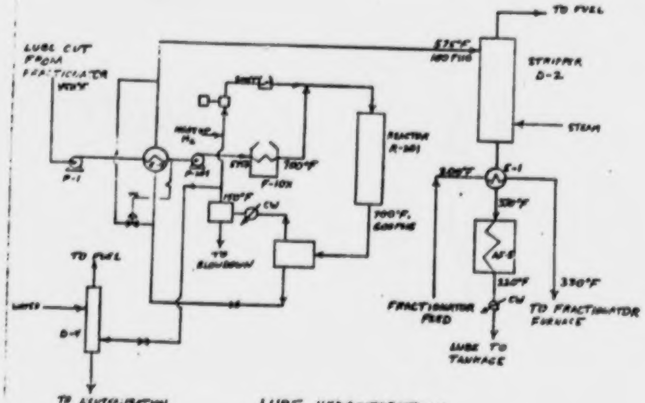

D-102
M-105
M-106
M-102
M-104
M-103
M-107
T-101
J-102
WATER
BUTANOL
BUTANOL - WATER
FEED
J-101
CENTRIFUGE
J-104
M-101
M-102
M-101
D-101
J-103
T-102
TREATED
OIL TO
VAC. DIST.

FIGURE 3-4


TO FUEL
LUBE CUT
FROM
FRACTIONATOR
575°F
STRIPPER
D-2
STEAM
REACTOR
R-101
P-1
P-101
F-101
700°F
E-1
TO FUEL
CW
TO
BLOWDOWN
700°F
WATER
D-4
220°F
330°F
CW
FRACTIONATOR
FEED
TO FRACTIONATOR
FURNACE
LUBE TO
TANKAGE
TO NEUTRALIZATION

LUBE HYDROTREATING
CASES B.1 AND C.1.

FIGURE 3-3

TABLE J-1

CASE DESCRIPTIONS

CASE	A	B.1.1.	B.1.2.	B.2.	C.1.	C.2.	C'.1.	C'.2.	D
FEED, MMGPY									
Crank Oil	9.0	9.0	9.0	39.0	10.8	34.8	10.8	34.8	29.0
Fuel Oil	-	-	30.0	-	-	-	-	-	-
SOLV. TREAT									
B/SD	-	-	-	-	1030	3322	1030	3322	-
Hrs./Yr.	-	-	-	-	6000	6000	6000	6000	-
VAC. FRACT.									
B/SD	1030	1030	3322	3322	1030	3322	1030	3322	3322
Hrs./Yr.	5000	5000	5000	5000	6000	6000	6000	6000	5000
HYDROFIN.									
B/SD	-	2115	2115	2115	-	-	2115	2115	2115
Hrs./Yr.	-	1350	1350	5000	-	-	2120	6750	5000
Feed, °F	-	50	450	450	-	-	50	450	450
	Present Oper.-Oper. Prob. -Poor Qual. Lube	Buy Exxon Monophin.	Buy Exxon Monophin. + Vac. Fract. Run Fuel Oil With Spare Cap.	Buy Exxon Monophin. + Vac. Fract.-Run Crank Oil Only	Add Solvent Treat	Add Solvent Treat + Exxon Vac. Fract.	Add Solvent Treat + Exxon Monophin.	Add Solvent Treat + Exxon Vac. Fract.+ Monophin.	Grass Roots Vac. Fract. + Hydrofin.
INCREMENTAL INVESTMENT, $M									
Exxon Equip	-	332	889	889	-	557	332	889	-
New Equip.	-	135	54	54	224	494	359	548	1,684
Util.	-	-	30	30	46	141	46	141	90
Waste	-	-	30	30	-	30	-	30	30
Tankage	-	-	95	95	28	140	28	140	746
Royalty	-	25	25	25	-	-	25	25	-
Offsites		492	1,123	1,123	298	1,362	750	1,773	2,550
									500
									3,050

TABLE J-2

PROFITS

CASE DESCRIPTION	A. Vac. Dist.		B.1.1. ---Vac. Dist. + Hydrofining---		B.1.2.		B.2.		C.1. Solv. Treat+Vac. Dist		C.2.		C'.1. Solvent Treating + Vac. Dist.		C'.2. +Hydrofin.		D Grass Roots(3)	
	MM GPY	¢/G	MM GPY	¢/G	MM GPY	¢/G	MM GPY	¢/G	MM GPY	¢/G	MM GPY	¢/G	MM GPY	¢/G	MM GPY	¢/G	MM GPY	¢/G
FEED																		
Crank Oil	9.0	3.00	9.0	3.00	9.0	3.00	29.0	3.00	10.8	3.00	34.8	3.00	10.8	3.00	34.8	3.00	34.8	3.00
Fuel Oil	-	-	-	-	20.0	2.00	-	-	-	-	-	-	-	-	-	-	-	-
Avg.					29.0	2.31												
OP. COSTS		4.83		6.26		2.24		2.45		4.84		2.22		6.22		2.90		2.75
FEED+OP.COSTS		7.83		9.26		4.55		5.45		7.84		5.22		9.22		5.90		5.75
NET PRODUCTS(1)																		
Lube Oil	5.75	10.22	5.75	10.22	5.75	1.86	18.52	10.22	7.76	11.51	25.00	11.51	7.76	11.51	25.00	11.51	18.52	10.22
Lt. Cuts	.35	0.39	.09	0.10	.06	.02	.82	.28	-	-	-	-	-	-	-	-	.82	.28
Bottoms	-	-	-	-	-	-	-	-	.59	.38	2.03	0.41	.23	0.15	1.52	0.31	-	-
Pb Sludge	1.96	0.38	1.96	0.38	1.96	0.12	6.34	0.38	.32	0.38	1.04	0.38	.32	0.38	1.04	0.38	6.34	.38
Fuel Oil	-	-	-	-	18.0	4.35	-	-	-	-	-	-	-	-	-	-	-	-
	8.06	10.99	7.80	10.70	25.77	6.35	25.68	10.88	8.67	12.27	28.07	12.30	8.31	12.04	27.56	12.20	25.68	10.88
PROFIT																		
¢/G Feed		3.16		1.44		1.80		5.43		4.43		7.08		2.82		6.30		5.13
$M/year.		285		129		522		1,573		478		2,462		303		2,194		1,488
RETURN																		
%/year B.T.(2)		-		26.2		46.5		140.1		160.3		180.7		38.4		123.8		48.8

(1) Excludes cuts used as fuel. Lube oil at 16¢/G; light cuts at 10¢/G; low metals bottoms at 7¢/G; Pb (lead) sludge at 10¢/lb. (o.5% Pb in crankcase oil - 100% recovery); fuel oil at 7¢/G.

(2) Return before taxes on incremental investment.

(3) Grass roots vacuum distillation plus hydrofining.

(4) Nomenclature: ¢/G = cents per gallon; MMGPY = millions of gallons per year; $M/year = thousands of dollars per year; %/year B.T. = percent per year before income tax.

TABLE J-3

SENSITIVITY ANALYSIS

CASE C'.2. (SOLVENT TREAT. + VAC. DIST. + HYDROFINING)

CASE	BASE (C^1.2)	1. DOUBLE PROCESS INVEST.	2. DECR. VOL. YIELD OF LUBE FROM 71.8% TO 63.9%	3. DECR. YIELD AS IN (2) AND NO BOTTOMS CREDIT	4. DECR. YIELD;NO BOTTOMS CREDIT; 8000 TO 5000 HRS.	5. DOUBLE OP. LABOR COST	6. DOUBLE INDIRECT COSTS	7. INCREASE FEED COST FROM 3 TO 5¢/G	8. INCREASE LUBE VALUE FROM 16 TO 20¢/G	9. INCREASE R$_1$ BY FACTOR OF 10	10. INCREASE SOLVENT LOST BY FACTOR OF 5
COSTS, ¢/G											
FEED	3.00	3.00	3.00	3.00	3.00	3.00	3.00	5.00	3.00	3.00	3.00
OPER.	2.90	3.46	2.86	2.86	3.43	3.42	3.38	2.90	2.90	4.06	4.55
	5.90	6.46	5.86	5.86	6.43	6.42	6.38	7.90	5.90	7.06	7.55
CREDITS, ¢/G	12.20	12.20	11.48	10.61	10.61	12.20	12.20	12.20	15.65	12.29	12.20
PROFIT											
¢/G	6.30	5.74	5.62	4.75	4.18	5.78	5.82	4.30	9.75	5.14	4.65
$MM/yr.	2.194	2.000	1.957	1.653	1.213	2.010	2.023	1.498	3.395	1.792	1.619
INCR. INV. $MM	1.773	3.322	1.773	1.773	1.773	1.773	1.773	1.773	1.773	1.773	1.773
RETURN %/yr. BT	123.8	60.3	110.2	93.4	68.4	113.2	114.1	84.4	191.3	101.0	91.3

Nomenclature: see Table 2.

TABLE J-4

COST BASES - YIELDS AND INVESTMENT

CASE DESCRIPTION	A VAC. DIST.	B.1.1.	B.1.2.	B.2.	C.1.	C.2.	C'.1.	C.'2.	D Grass Roots (1)
		Vac. Dist. + Hydrof.			Solv. Treat + Vac. Dist.		Solv. Treat + Vac. Dist. + Hydrof.		
FEED, MMGPY									
Crank Fuel	9.0	9.0	9.0	29.0	10.0	34.0	10.0	34.0	29.0
Fuel Oil	-	-	20.0	-	-	-	-	-	-
YIELD, MMGPY									
Lube	5.75	5.75	5.75	18.52	7.76	25.00	7.76	25.00	18.52
Lt. Cuts	0.94	0.94	0.94	3.02	1.13	3.62	1.13	3.62	3.02
Bot.	-	-	-	-	1.18	3.81	1.18	3.81	-
Pb. Sludge	1.96	1.96	1.96	6.34	0.32	1.04	.32	1.04	6.34
Fuel Oil	-	-	18.00	-	-	-	-	-	-
	8.65	8.65	26.65	27.88	10.39	33.47	10.39	33.47	27.88
PLANT VALUE $M (2)									
Process	500	1264	1738	1738	724	1949	1488	2232	1684
Util + Waste	70	70	120	120	116	231	116	231	120
Tankage	651	651	746	746	679	857	679	857	746
	1221	1985	2604	2604	1519	3037	2283	3320	2550
INCR. INV. $M (2)	-	492	1123	1123	298	1362	790	1773	2050

(1) Vacuum distillation plus hydrofining
(2) Excludes land and operating capital; Incr. Inv. = incremental investment

TABLE J-5

COSTS (1)

CASE DESCRIPTION	A. Vac. Dist.	B.1.1. ---Vac. Dist. + Hydrofining---	B.1.2.	B.2.	C.1. Solv. Treat+Vac. Dist	C.2.	C'.1. Solv. Treat. + Vac Dist + Hydrofining	C'.2.	D Grass Roots
FEED, MMGPY									
Crank Oil	9.0	9.0	9.0	29.0	10.8	34.0	10.8	34.0	29.0
Fuel Oil	-	-	20.0	-	-	-	-	-	-
PV,$MM	1.221	1.985	2.604	2.604	1.519	2.837	2.303	3.330	2.500
Incr. Inv. $MM	-	0.492	1.133	1.133	0.290	1.362	0.790	1.773	3.050
Depr. Base, $MM	1.221	1.713	1.844	1.844	1.519	2.083	2.011	2.494	3.050
DIR. OP. COSTS,$M/Yr.									
Op. Labor	90.0	100.0	110.0	100.0	100.0	110.0	120.0	130.0	100.0
Labor O.H.	36.0	40.0	44.0	40.0	40.0	44.0	48.0	52.0	40.0
Ins. + Taxes	36.6	59.6	78.2	78.2	45.6	79.1	60.5	99.7	76.6
Catalyst	-	14.7	14.7	47.3	-	-	19.9	64.0	67.3
N-Butanol	-	-	-	-	19.7	63.9	19.7	63.9	-
Antifoulant	-	2.3	2.3	7.3	-	-	-	-	7.3
Hydrogen	-	10.3	10.3	26.5	-	-	13.9	44.7	26.5
Maint.	36.6	59.6	78.2	78.2	45.6	79.7	60.5	99.7	76.6
Deprec.	81.5	114.3	123.1	123.1	101.3	139.2	134.1	166.3	203.5
Power	10.6	19.0	23.1	53.6	25.9	83.3	34.0	114.0	53.6
City Water	1.2	1.7	2.0	5.2	2.0	6.2	2.4	7.8	5.2
	292.5	421.5	485.9	559.4	380.1	605.4	529.0	842.1	656.6
¢/G Feed	3.25	4.68	1.67	1.93	3.52	1.78	4.90	2.48	2.23
INDIR. OP. COSTS$MM/Yr.									
Salaries	80.0	80.0	90.0	80.0	80.0	90.0	80.0	90.0	80.0
Salary O.H.	32.0	32.0	36.0	32.0	32.0	36.0	32.0	36.0	32.0
Lab + Office Exp.	10.0	10.0	15.0	15.0	10.0	15.0	10.0	15.0	15.0
Other	20.0	20.0	25.0	25.0	20.0	25.0	20.0	25.0	25.0
	142.0	142.0	166.0	152.0	142.0	166.0	142.0	166.0	152.0
¢/G Feed	1.58	1.58	0.57	0.52	1.32	0.48	1.32	0.48	0.52

(1) See Table 6 for cost bases. Nomenclature - see Table 2.

(2) Later information indicated hydrogen consumption about ten times this value. See Appendix E.

TABLE J-6
BASES FOR OPERATING COSTS

Operating Labor - $10,000 wages per man per year.

Labor Overhead - direct overhead @ 40% of operating labor.

Insurance & taxes - insurance and local property taxes at 3% of plant value. (See table 4.)

Catalyst - $1.10 per pound; one year life for cases B.2. and D.; other cases prorated on the basis of feed thruput.

n-Butanol - 10¢/lb.

Anti-foulant - 100 ppm (volume basis) @ $2.50 per gallon.

Hydrogen - $4.00 per 1000 SCF delivered; 15 SCF/B of actual throughput used.*

Maintenance - 3% of plant value per year (assuming routine maintenance done by operating personnel.)

Depreciation - 6.67%/year of capital invested in usable equipment.

Power - 3.5 to 4.0 ¢/KWH depending upon usage.

City water - 70¢ per thousand gallons.

*Later information indicated that hydrogen consumption was probably closer to 150 SCF/B. See Appendix E.

Table K-1. NOVEMBER 1972 WASTE FUEL OIL RUN
Run Length = 51 hrs.

	Feed	Water	Overhead	Bottoms
Total gallons	176,283	12,299	7,000	156,984
Yield, %	--	6.95	3.95	89.1
Gals./hr.	3,455	241	137	3,077
Gravity, °API	25.2	10.0	41.0	26.0
lbs./hr.	25,960	2,014	935	23,020
BTU/lb. furnace input (calculated)	--	1,118	137	73
Fired Heater Duty, BTU/hr. (Total = 4,055,100)	--	2,247,000	128,100	1,680,000

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Table K-2. DECEMBER 1972 WASTE FUEL OIL RUNS
(Averages for two runs)

Heater inlet temperature	110°F	
Heater transfer line	215°F	
Fractionator bottom	182°F	
Fractionator top	175°F	
Heater stack	625°F	
Heater flue gas recirculated	520°F	
Bottom of fractionator vacuum	25.2 in. Hg.	
Heater inlet pressure	46 psig	
Feed gravity	23.2°API	
Bottoms product gravity	25.3°API	
Feed to heater	2714 gal./hr.	
Bottoms product	2235 gal./hr.	(82.42%)
Overhead	81 gal./hr.	(2.99%)
Water	374 gal./hr	(13.71%)
Loss	24 gal./hr	(0.88%)

Table K-3. JANUARY 1973 WASTE FUEL OIL RUN

Start: 1/4/73, 11:30 AM Stop: 1/5/73, 2:45 AM
On Stream: 15 hrs., 15 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2705	41125		23.8	3.62
Bttms	2533	38628	No. 6 Fuel Oil	25.7	Trace
Overhead	39	595		40.2	Trace
Water & Loss	133	1902			

STREAMS	FLOW
Cooling Water	350 GPM
Steam Produced	960 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	320 " "
Steam to Tanks & Line Loss	184 " "

TEMPERATURES LOCATION	°F
Oil H't'r Inlet	115
Oil H't'r Outlet	218
Fractionator:	
Bottom	180
Top	175
Flash Zone	180

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	49 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	24.1 IN.HG Vacuum
Steam Boilers		

Table K-4. JANUARY 1973 WASTE FUEL OIL RUN

Start: 1/8/73, 12:10 PM Stop: 1/9/73, 3:15 PM
On Stream: 27 hrs., 5 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2266	61400		23.0	6.61
Bttms	2092	56650	No. 6 Fuel Oil	25.5	Trace
Overhead	20	542		41.0	Trace
Water & Loss	154	4208			

STREAMS	FLOW
Cooling Water	350 GPM
Steam Produced	960 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	320 " "
Steam to Tanks & Line Loss	184 " "

TEMPERATURES LOCATION	°F
Oil H't'r Inlet	116
Oil H't'r Outlet	217
Fractionator:	
Bottom	183
Top	175
Flash Zone	185

EQUIPMENT	LOCATION	UNITS
Furnace Coil	Inlet	47 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	24.3 IN. HG Vacuum
Steam Boilers		

Table K-5. JANUARY 1973 WASTE FUEL OIL RUN

Start: Stop:
On Stream: 16 hrs., 30 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2242	37060			
Bttms	1795	29640	No. 6 Fuel Oil		
Overhead	45	743			
Water & Loss	402	6677			

STREAMS	FLOW
Cooling Water	350 GPM
Steam Produced	960 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	320 " "
Steam to Tanks & Line Loss	184 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	120
Oil H't'r Outlet	219
Fractionator:	
Bottom	181
Top	173
Flash Zone	185

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	53 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	24.7 IN. HG Vacuum
Steam Boilers		

Table K-6. JANUARY 1973 WASTE FUEL OIL RUN

Start: 1/15/73, 1:45 PM Stop: 1/16/73, 8:30 AM
On Stream: 18 hrs., 15 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2017	37818		19.7	17.45
Bttms	1593	29961	No. 6 Fuel Oil	25.6	Trace
Overhead	62	1162		14.0	Trace
Water & Loss	362	6695			

STREAMS	FLOW
Cooling Water	350 GPM
Steam Produced	960 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	320 " "
Steam to Tanks & Line Loss	184 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	117
Oil H't'r Outlet	213
Fractionator:	
Bottom	180
Top	176
Flash Zone	180

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	57 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	25.7 IN.HG Vacuum
Steam Boilers		

Table K-7. JANUARY 1973 WASTE FUEL OIL RUN

Start: 1/17/73, 12:40 PM Stop: 1/18/73, 3:45 AM
On Stream: 15 hrs., 5 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2530	38197		21.5	10.74
Bttms	1854	27993	No. 6 Fuel Oil	25.9	Trace
Overhead	383	5780		41.5	Trace
Water & Loss	293	4424			

STREAMS	FLOW
Cooling Water	350 GPM
Steam Produced	960 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	" "
Steam to Tanks & Line Loss	" "

TEMPERATURES LOCATION	°F
Oil H't'r Inlet	120
Oil H't'r Outlet	217
Fractionator:	
Bottom	182
Top	176
Flash Zone	183

EQUIPMENT	LOCATION	UNITS
Furnace Coil	Inlet	51 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	25.1 IN.HG Vacuum
Steam Boilers		

Table K-8. JANUARY 1973 WASTE FUEL OIL RUN

Start: 1/24/73, 8:55 AM Stop: 1/26/73, 7:00 AM
On Stream: 46 hrs., 5 min.
Feedstock type: Tank bottoms, tank washings, [illegible]
containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2215	102100		20.0	18.15
Bttms	1580	72806		23.0	Trace
Overhead	215	9900		39.9	Trace
Water & Loss	420	19394			

		TEMPERATURES	
STREAMS	FLOW	LOCATION	°F
Cooling Water	350 GPM	Oil H't'r Inlet	115
Steam Produced	960 LBS/HR	Oil H't'r Outlet	230
Steam for Stripping	None " "	Fractionator:	
Steam to Vacuum Jets	456 " "	Bottom	190
Steam to Tower	None " "	Top	179
Steam Bttms Pump	200 " "	Flash Zone	190
Steam to Tanks & Line Loss	314 " "		

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	62 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	24.0 IN.HG Vacuum
Steam Boilers		

Table K-9. JANUARY/FEBRUARY WASTE FUEL OIL RUN

Start: 1/31/73, 11:00 AM Stop: 2/1/73, 12:30 PM
On Stream: 25 hrs., 30 Min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	1503	38385		17.5	11.37
Bttms	973	24820	No. 6 Fuel Oil	19.8	Trace
Overhead	343	8650		39.5	Trace
Water & Loss	187	4915			

STREAMS	FLOW
Cooling Water	350 GPM
Steam Produced	870 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	446 " "
Steam to Tower	None " "
Steam Bttms Pump	170 " "
Steam to Tanks & Line Loss	264 " "

TEMPERATURES LOCATION	°F
Oil H't'r Inlet	118
Oil H't'r Outlet	220
Fractionator:	
Bottom	195
Top	180
Flash Zone	190

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	35 PSIG
Furnace Coil	Outlet	---PSIG
Fractionator	Flash Zone	24.7 IN.HG Vacuum
Steam Boilers		

Table K-10.FEBRUARY 1973 WASTE FUEL OIL RUN

Start: 2/7/73, 3:15 PM Stop: 2/8/73, 9:00PM
On Stream: 17hrs, 45 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2116	100.0	37596	No.6 Fuel Oil	14.1	15.1
Bttms	1662	78.4	29500		22.8	Trace
Overhead	133	6.3	2360		44.3	Trace
Water & Loss	321	15.3	5736			

STREAMS	FLOW		TEMPERATURES	
			LOCATION	°F
Cooling Water	180	GPM	Oil H't'r Inlet	117
Steam Produced	946	LBS/HR	Oil H't'r Outlet	195
Steam for Stripping	None	" "	Fractionator:	
Steam to Vacuum Jets	456	" "	Bottom	184
Steam to Tower	None	" "	Top	178
Steam Bttms Pump	320	" "	Flash Zone	180
Steam to Tanks & Line Loss	170	" "		

EQUIPMENT	LOCATION	UNITS
Furnace Coil	Inlet	59 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	22.4 In. HG Vacuum
Steam Boilers		

Table K-11.FEBRUARY 1973 WASTE FUEL OIL RUN

Start: 2/14/73, 11:30 AM Stop: 2/15/73, 10:00 AM
On Stream: 22 hrs., 30 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	1636	100.0	36818		18.3	26.0
Bttms	1265	77.3	28464		22.8	Trace
Overhead	26	1.6	584		42.1	Trace
Water & Loss	345	21.1	7770			

STREAMS	FLOW
Cooling Water	180 GPM
Steam Produced	906 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	280 " "
Steam to Tanks & Line Loss	170 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	118
Oil H't'r Outlet	205
Fractionator:	
Bottom	178
Top	175
Flash Zone	180

EQUIPMENT	LOCATION	UNITS
Furnace Coil	Inlet	53 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	25.0 In. HG Vacuum
Steam Boilers		

Table K-11. FEBRUARY 1973 WASTE FUEL OIL RUN (Continued)

Start: 2/14/73, 11:30 AM Stop: 2/15/73, 10:00 AM

MATERIAL	FEED		BOTTOMS	OVERHEAD
°API	18.3		22.8	42.1
Distillation	Still	Vapor		
IBP °F	212	100	264	
5% Recovered	-	-	455	
10%	440	205	539	
20	600	358	635	
30	652	454	650	
40	678	462	651	
50	697*	457*	652	
60	712	458	655	
70			657	
80			658*	
90				
H_2O Content%	26.0		Trace	Trace
Recovery, %			89	

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* Cracked

Table K-12.

FEBRUARY 1973 WASTE FUEL OIL RUN

Start: 2/21/73, 10:30 AM Stop: 2/23/73, 6:00 PM
On Stream: 55 hrs., 30 Min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2084	100.0	115725		21.3	13.6
Bttms	1761	84.5	97800	No.6 Fuel Oil	24.7	Trace
Overhead	39	1.9	2162		40.0	Trace
Water & Loss	284	13.6	15763			

STREAMS	FLOW
Cooling Water	180 GPM
Steam Produced	931 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	310 " "
Steam to Tanks & Line Loss	165 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	115
Oil H't'r Outlet	205
Fractionator:	
Bottom	187
Top	183
Flash Zone	184

EQUIPMENT	LOCATION	UNITS
Furnace Coil	Inlet	54 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	25.7 In. HG Vacuum
Steam Boilers		

Table K-13.

FEBRUARY 1973 WASTE FUEL OIL RUN

Start: 2/26/73, 3:35 PM Stop: 2/27/73, 8:10 PM
On Stream: 28 hrs., 35 Min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	1461	100.0	41738		24.1	7.4
Bttms	996	68.2	28464	No.6 Fuel Oil	24.9	Trace
Overhead	123	8.4	3518		40.4	Trace
Water & Loss	342	23.4	9756			

STREAMS	FLOW	TEMPERATURES	
		LOCATION	°F
Cooling Water	170 GPM	Oil H't'r Inlet	116
Steam Produced	836 LBS/HR	Oil H't'r Outlet	220
Steam for Stripping	None " "	Fractionator:	
Steam to Vacuum Jets	456 " "	Bottom	185
Steam to Tower	None " "	Top	180
Steam Bttms Pump	220 " "	Flash Zone	183
Steam to Tanks & Line Loss	160 " "		

EQUIPMENT	LOCATION	UNITS
Furnace Coil	Inlet	18 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	26.8 IN.HG Vacuum
Steam Boilers		

Table K-14.

MARCH 1973 WASTE FUEL OIL RUN

Start: 3/5/73, 1:30 PM **Stop:** 3/6/73, 8:30 AM
On Stream: 19 hrs.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	1906	36224		20.2	30.0
Bttms	1117	21244	No. 6 Fuel Oil	24.4	-
Overhead	139	2641		41.4	-
Water & Loss	650	12,339			

STREAMS	FLOW
Cooling Water	180 GPM
Steam Produced	806 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	180 " "
Steam to Tanks & Line Loss	170 " "

TEMPERATURES LOCATION	°F
Oil H't'r Inlet	122
Oil H't'r Outlet	210
Fractionator:	
Bottom	181
Top	175
Flash Zone	182

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	63 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	25.3 IN.HG Vacuum
Steam Boilers		

Table K-14.

MARCH 1973 WASTE FUEL OIL RUN (Continued)

Start: 3/5/73, 1:30 PM Stop: 3/6/73, 8:30 AM

MATERIAL	FEED		BOTTOMS	OVERHEAD
°API	20.2		24.4	41.4
Distillation	°F		°F	°F
IBP	208	Water 30.0%	460	190
5% Recovered	208		500	240
10%	210		560	264
20	211		657	290
30	213		660	308
40	426		677	328
50	608		678	344
60	689		690	358
70	714		696	376
80	724		693	396
90	716	(87%)		461
FBP	716			526
% Recovery	87.0		85.0	99%
Water Content %	30.0		Trace	Trace

Table K-15. MARCH 1973 WASTE FUEL OIL RUN

Start: 3/7/73, 5:45 PM **Stop:** 3/8/73, 6:00 AM
On Stream: 12 hrs., 15 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2880	35320		21.4	28.1
Bttms	1860	22780	No. 6 Fuel Oil	25.9	Trace
Overhead	139	2628		41.7	Trace
Water & Loss	881	9912			

STREAMS	FLOW
Cooling Water	180 GPM
Steam Produced	836 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	210 " "
Steam to Tanks & Line Loss	170 " "

TEMPERATURES LOCATION	°F
Oil H't'r Inlet	118
Oil H't'r Outlet	212
Fractionator:	
Bottom	200
Top	195
Flash Zone	197

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	56 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	25.5 IN.HG Vacuum
Steam Boilers		

Table K-16. MARCH 1973 WASTE FUEL OIL RUN

Start: 3/15/73, 11:15 PM **Stop:** 3/16/73, 5:30 PM
On Stream: 18 hrs., 45 min.
Feedstock type: **Tank bottoms, tank washings, fuel oil containing water, solids, gasoline**

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	1963	35830		20.7	35.5
Bttms	1122	20496	No. 6 Fuel Oil	25.6	Trace
Overhead	142.3	2602		41.4	Trace
Water & Loss	699	12732			

STREAMS	FLOW
Cooling Water	180 GPM
Steam Produced	846 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	230 " "
Steam to Tanks & Line Loss	160 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	118
Oil H't'r Outlet	218
Fractionator:	
Bottom	186
Top	180
Flash Zone	188

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	59 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	24.6 IN.HG Vacuum
Steam Boilers		

Table K-17. MARCH 1973 WASTE FUEL OIL RUN

Start: 3/21/73, 11:50 AM Stop: 3/22/73, 2:00 PM
On Stream: 26 hrs., 10 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	3820	69505		23.0	11.3
Bttms	2158	56400	No. 6 Fuel Oil	26.4	Trace
Overhead	205	5250		41.5	Trace
Water & Loss	1457	7855			

STREAMS	FLOW
Cooling Water	130 GPM
Steam Produced	876 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	260 " "
Steam to Tanks & Line Loss	160 " "

TEMPERATURES LOCATION	°F
Oil H't'r Inlet	119
Oil H't'r Outlet	214
Fractionator:	
Bottom	182
Top	178
Flash Zone	185

EQUIPMENT	LOCATION	UNITS
Furnace Coil	Inlet	51 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	23.0 IN. HG Vacuum
Steam Boilers		

Table K-18. **MARCH 1973 WASTE FUEL OIL RUN**

Start: 3/26/73, 10:40 AM **Stop:** 3/27/73, 6:30 AM
On Stream: 19 hrs., 50 min.
Feedstock type: **Tank bottoms, tank washings, fuel oil containing water, solids, gasoline**

Streams	**Flow Rate Gal/hr.**	**Volume** Gallons	Sold As	Gravity °API	**Water %**
Feed	2509	50633		22.3	7.1
Bttms	2213	44600	No. 6 Fuel Oil	26.4	Trace
Overhead	121.3	2446		41.6	Trace
Water & Loss	175	3587			

STREAMS	**FLOW**	TEMPERATURES LOCATION	°F
Cooling Water	120 GPM	Oil H't'r Inlet	120
Steam Produced	876 LBS/HR	Oil H't'r Outlet	210
Steam for Stripping	None " "	Fractionator:	
Steam to Vacuum Jets	456 " "	Bottom	182
Steam to Tower	None " "	Top	178
Steam Bttms Pump	260 " "	Flash Zone	185
Steam to Tanks & Line Loss	160 " "		

EQUIPMENT LOCATION		UNITS
Furnace Coil	**Inlet**	52 PSIG
Furnace Coil	**Outlet**	- PSIG
Fractionator	**Flash Zone**	21.9 IN.HG Vacuum
Steam Boilers		

Table K-19. **MARCH 1973 WASTE FUEL OIL RUN**

Start: 3/28/73, 10:30 AM **Stop:** 3/29/73, 3:00 AM
On Stream: 16 hrs., 30 min.
Feedstock type: **Tank bottoms, tank washings, fuel oil containing water, solids, gasoline**

Streams	Flow Rate Gal/hr.	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2435	40146	No. 6 Fuel Oil	22.8	7.3
Bttms	2135	35206		26.3	Trace
Overhead	121.0	1996		41.7	Trace
Water & Loss					
Total					

STREAMS	FLOW
Cooling Water	120 GPM
Steam Produced	886 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	260 " "
Steam to Tanks & Line Loss	170 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	117
Oil H't'r Outlet	212
Fractionator:	
Bottom	180
Top	175
Flash Zone	176

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	54 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	24.6 IN.HG Vacuum
Steam Boilers		

Table K-20. APRIL 1973 WASTE FUEL OIL RUN

Start: 4/2/73, 10:25 AM Stop: 4/2/73, 11:45 PM
On Stream: 13 hrs., 10 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	3225	100.0	42400		24.3	
Bttms	2785	86.7	36720	No. 6 Fuel Oil	26.3	
Overhead	175	5.2	2220		41.2	
Water & Loss	265	8.1	3460			

STREAMS	FLOW
Cooling Water	130 GPM
Steam Produced	896 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	280 " "
Steam to Tanks & Line Loss	160 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	116
Oil H't'r Outlet	219
Fractionator:	
Bottom	193
Top	190
Flash Zone	191

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	57 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	26.3 IN.HG Vacuum
Steam Boilers		110 PSIG

Table K-21. APRIL 1973 WASTE FUEL OIL RUN

Start: 4/9/73, 10:25 AM Stop: 4/10/73, 1:55 AM
On Stream: 13 hrs., 55 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2704	100.0	37600	No. 6 Fuel Oil	22.0,18.2	
Bttms	2235	82.6	31046		26.0,22.9	
Overhead	195	7.2	2710		40.2,43.0	
Water & Loss	274	10.2	3844			

		TEMPERATURES	
STREAMS	FLOW	LOCATION	°F
Cooling Water	125 GPM	Oil H't'r Inlet	116
Steam Produced	816 LBS/HR	Oil H't'r Outlet	210
Steam for Stripping	None " "	Fractionator:	
Steam to Vacuum Jets	456 " "	Bottom	180
Steam to Tower	None " "	Top	175
Steam Bttms Pump	190 " "	Flash Zone	176
Steam to Tanks & Line Loss	170 " "		

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	60 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	23.7 IN. HG Vacuum
Steam Boilers		110 PSIG

Table K-22. **APRIL** 1973 WASTE FUEL OIL RUN

Start: **4/10/73, 4:50 PM** Stop: **4/11/73, 9:30 AM**
On Stream: **14 hrs., 30 min.**
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2877	100.0	41660		21.6	
Bttms	2527	87.9	36620	No. 6 Fuel Oil	25.5	
Overhead	138	4.8	2000		41.0	
Water & Loss	212	7.3	3040			

STREAMS	FLOW
Cooling Water	125 GPM
Steam Produced	816 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	200 " "
Steam to Tanks & Line Loss	160 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	118
Oil H't'r Outlet	225
Fractionator:	
Bottom	180
Top	175
Flash Zone	175

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	61 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	23.5 IN.HG Vacuum
Steam Boilers		110 PSIG

Table K-23. **APRIL 1973 WASTE FUEL OIL RUN**

Start: 4/16/73, 11:45 AM Stop: 4/17/73, 9:00 AM
On Stream: 21 hrs., 15 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	1670		35250		19.3	
Bttms	1012	60.63	21379	No.6 Fuel Oil	24.5	
Overhead	51	3.05	1076		39.4	
Water & Loss	609	36.32	12795			

STREAMS	FLOW
Cooling Water	150 GPM
Steam Produced	766 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	150 " "
Steam to Tanks & Line Loss	160 " "

TEMPERATURES LOCATION	°F
Oil H't'r Inlet	117
Oil H't'r Outlet	210
Fractionator:	
Bottom	182
Top	178
Flash Zone	180

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	58 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	24.4 IN.HG Vacuum
Steam Boilers		110 PSIG

Table K-24. APRIL 1973 WASTE FUEL OIL RUN

Start: 4/18/73, 1:30 PM Stop: 4/19/73, 3 PM
On Stream: 25 hrs., 30 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	1491	100.00	38014		18.9	
Bttms	1316	88.24	33561	No.6 Fuel Oil	23.3	
Overhead	67	4.50	1706		4..0	
Water & Loss	108	7.26	2747			

STREAMS	FLOW
Cooling Water	110 GPM
Steam Produced	746 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	140 " "
Steam to Tanks & Line Loss	150 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	121
Oil H't'r Outlet	216
Fractionator:	
Bottom	185
Top	175
Flash Zone	182

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	42 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	26.3 IN.HG Vacuum
Steam Boilers		110 PSIG

Table K-25. **APRIL** 1973 WASTE FUEL OIL RUN

Start: 4/25/73, 1:20 PM Stop: 4/26/73, 1:00 PM
On Stream: 23 hrs., 40 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Water %
Feed	2153	100.00	48622		
Bttms	1898	88.15	42864	No. 6 Fuel Oil	
Overhead	95	4.40	2138		
Water & Loss	160	7.45	3620		

STREAMS	FLOW
Cooling Water	110 GPM
Steam Produced	791 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	185 " "
Steam to Tanks & Line Loss	150 " "

TEMPERATURES LOCATION	°F
Oil H't'r Inlet	118
Oil H't'r Outlet	215
Fractionator:	
Bottom	182
Top	175
Flash Zone	182

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	59 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	24.4 IN.HG Vacuum
Steam Boilers		110 PSIG

Table K-25.

APRIL 1973 WASTE FUEL OIL RUN (Continued)

Start: 4/25/73, 1:20 PM Stop: 4/26/73, 1:00 PM

MATERIAL	FEED	BOTTOMS	OVERHEAD
°API	19.9	24.5	36.8
Distillation	°F	°F	°F
IBP	268	290	210
5% Recovered	318	500	278
10%	385	545	290
20	556	610	221
30	570	654	360
40	614	668	380
45	606	-	-
50	-	654	395
60			412
70			432
80			457
90			490
FBP	606	654	528
% Recovery	45	50.0	96.0

Table K-26.

APRIL/MAY 1973 WASTE FUEL OIL RUN

Start: 4/30/73, 1:40 PM Stop: 5/1/73, 12:00 Noon
On Stream: 22 hrs., 20 min.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2006	100.00	44850		17.4	
Bttms	1107	55.24	24774		22.6	
Overhead	144	7.21	3236		39.8	
Water & Loss	755	37.55	16840			

STREAMS	FLOW
Cooling Water	210 GPM
Steam Produced	751 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	150 " "
Steam to Tanks & Line Loss	145 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	110
Oil H't'r Outlet	212
Fractionator:	
Bottom	181
Top	178
Flash Zone	180

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	60 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	24.0 IN.HG Vacuum
Steam Boilers		110 PSIG

Table K-27. MAY 1973 WASTE FUEL OIL RUN

Start: 5/24/73, 1:30 PM Stop: 5/30/73, 10:20 AM
On Stream: 20.833
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	1673	100.00	34848			
Bttms	1118.4	66.85	23303	No. 6 Fuel Oil		
Overhead	54.0	3.23	1126			
Water & Loss	560.6	29.92	10419			

STREAMS	FLOW
Cooling Water	160 GPM
Steam Produced	746 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	150 " "
Steam to Tanks & Line Loss	140 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	116
Oil H't'r Outlet	216
Fractionator:	
Bottom	181
Top	178
Flash Zone	184

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	56 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	24.7 IN.HG Vacuum
Steam Boilers		105 PSIG

Table K-27.

MAY 1973 WASTE FUEL OIL RUN (Continued)

Start: 5/24/73, 1:30 PM Stop: 5/30/73, 10;20 AM

MATERIAL	FEED	BOTTOMS	OVERHEAD
°API	19.3	23.5	36.9
Distillation	°F	°F	°F
IBP	269	291	187
5% Recovered	399	364	214
10%	500	414	237
20	566	506	285
30	589	554	350
40	602	584	418
50	615	601 (64%)	452
60	620		503
70	612 (65%)		529
80			540
90			540 (85%)
FBP	620	601	540
% Recovery	65	46	85

Table K-28. **MAY** 1973 WASTE FUEL OIL RUN

Start: 5/31/73, 10:30 AM Stop: 5/31/73, 6:30 PM
On Stream: 8.00 hrs.
Feedstock type: Tank bottoms, tank washings, fuel oil containing water, solids, gasoline

Streams	Flow Rate Gal/hr.	Volume Per Cent	Volume Gallons	Sold As	Gravity °API	Water %
Feed	2986.5	100.0	23892		23.9	
Bttms	2692	90.18	21540	No. 6 Fuel Oil	25.8	
Overhead	98	3.1	744		37.7	
Water & Loss	201	6.72	1608			

STREAMS	FLOW
Cooling Water	125 GPM
Steam Produced	806 LBS/HR
Steam for Stripping	None " "
Steam to Vacuum Jets	456 " "
Steam to Tower	None " "
Steam Bttms Pump	205 " "
Steam to Tanks & Line Loss	145 " "

TEMPERATURES	
LOCATION	°F
Oil H't'r Inlet	122
Oil H't'r Outlet	222
Fractionator:	
Bottom	183
Top	180
Flash Zone	185

EQUIPMENT LOCATION		UNITS
Furnace Coil	Inlet	56 PSIG
Furnace Coil	Outlet	- PSIG
Fractionator	Flash Zone	24.1 IN.HG Vacuum
Steam Boilers		105 PSIG

APPENDIX L

MANUFACTURER'S DATA ON HYDRIDES

10-71 1M
BULLETIN NO. 2-A

SODIUM BOROHYDRIDE

Formula:	$NaBH_4$
Molecular Weight:	37.85
Specific gravity:	1.074 g./cc. at 25°C.
Color:	White

Functional Groups Reduced by $NaBH_4$:

Sodium borohydride rapidly reduces most aldehydes, ketones, peroxides and hydroperoxides. It reduces, at a slower rate, Schiff bases and cyclic quaternary iodides. Under special conditions, e.g., through the use of Lewis acids such as aluminum chloride, its reducing power can be enhanced so that it is almost comparable to lithium aluminum hydride.

See brochure on Sodium Borohydride for additional data.

Suggested Uses:

Sodium borohydride has been used extensively in the pharmaceutical and fine flavor fields for many years, for the conversion of aldehydes and ketones to the corresponding alcohols. It has found use as a polymerization catalyst,[1] as a blowing agent for various plastic and rubber foams,[2] for removal of trace impurities in many different process impurities in many different process streams, [3] and for hydrogen generation.[4] Ventron technical bulletins are available in each specific area. Other uses include metal plating and catalyst preparation.

Availability:

Sodium borohydride may be purchased as a dry powder or as pellets in 10/32" and 24/32" sizes. A third form, a stabilized water solution (SWS), is also available.

Sodium borohydride powder and pellets can be supplied in quantities varying from 100 grams to car loads, while SWS is available in one gallon to tank car lots.

Thermal Stability:

Decomposition starts without melting at above 400°C. in dry air.

Solubility at 25°C.:

Solvent	**g./100 g. solvent**	
liquid ammonia	104.0	
water	55.0	
ethanol	4.0	(reacts slowly)
dimethyl ether of diethylene glycol	5.5	
dimethyl formamide	18.0	
isopropylamine	6.0	

For additional solubility data see Sodium Borohydride brochure (available on request).

Typical Assay:

$NaBH_4$	98.0%

Packaging and Shipping:

Sodium borohydride is shipped in polyethylene bags in metal containers. Shipment is governed by I.C.C. regulations 73:153, 73:154 and 73:206. Unlimited quantities can be shipped by Railway Express or Motor Freight.

Handling and Storage:

Sodium borohydride may be handled in air according to safe practice for inflammable hygroscopic powders (comparable to calcium carbide). The unconfined powder does not ignite on contact with moisture, but forms a dihydrate which slowly hydrolyzes. It is stable to shock.

Safety:

Both dry borohydrides and their aqueous solutions are relatively safe materials to handle. Certain precautions must be taken, however. Borohydride solutions, if overheated, subjected to acid conditions, or in the presence of the metal salts or finely divided metallic precipitates of nickel, cobalt, or iron, will decompose rapidly, evolving large amounts of hydrogen. Other than this, treat as flake caustic or a 50% caustic solution.

Toxicity:

Sodium borohydride is considered toxic if ingested primarily due to possible gas embolism resulting from reaction with stomach acids. All precautions should be taken against ingestion, inhalation of dust, or contact with skin.

First Aid:

In case of accidental contact with the skin, the particles should be brushed off and the affected area flooded with water.

References:

Ventron Corporation Technical Bulletins:

1. Borohydrides in Polymerization Processes.
2. Sodium Borohydride for Flexible Polyvinyl Chloride Sponge.
3. Process Stream Purification Through Hydride Chemistry.
4. Metal Hydrides for Hydrogen Generation.

The information contained in this bulletin is, to our best knowledge, true and accurate. Since the conditions of use are beyond our control, we assume no obligation or liability in connection therewith. Nothing in this bulletin shall be construed as permission or recommendation to practice a patented invention without a license.

Ventron Corporation/Chemicals Division
Congress Street, Beverly, Massachusetts 01915/Tel: (617) 922-1875

Sodium Borohydride-SWS*

(*Stabilized Water Solution)

Sodium Borohydride-SWS is a stable, alkaline aqueous solution of sodium borohydride, which greatly facilitates the application of sodium borohydride in chemical processing, particularly to water-based systems. Its handling characteristics closely resemble those of 50% liquid caustic.

For information on uses, see brochure on Sodium Borohydride.

Composition:	$NaBH_4$	12 ± 0.5%
	NaOH	42 ± 2%
	H_2O	Balance

Specific Gravity: 1.4 g/ml. or 11.7 lbs./gal.

Color and Form: Off-white Liquid

Viscosity: 79.0 centipoise at 23°C.

Stability: $NaBH_4$-SWS is remarkably stable under normal storage and shipping conditions. It decomposes very slowly according to the equation:

$$NaBH_4 + 2H_2O \rightarrow NaBO_2 + 4H_2$$

The rate varies slightly with temperature. Typical rates are:

t(°C.)	% decomp. per day
21	0.000005
54	0.0002
100	0.008

Solubility:

Sodium Borohydride-SWS can be diluted for use with water or methanol. Many organic solvents normally miscible with water are not suitable because of the caustic present.

Handling and Storage:

Sodium Borohydride-SWS is similar to the 50% liquid caustic which is an article of commerce. Accepted storage and handling procedures for 50% liquid caustic apply also for Sodium Borohydride-SWS. On long standing, a pressure may build up over the solution. Containers should be periodically checked. There should be at least 10% free volume in all closed containers. If this rule is followed, pressure build-up will be less than 1 psi per year at ambient room temperature. Some suitable container materials are stainless steels, mild steel, and polyethylene. Glass is attacked by the strong caustic. Sodium Borohydride-SWS should be stored at temperatures above 65°F. Sodium Borohydride-SWS becomes more viscous below 60°F. and can crystallize at temperatures below 55°. To liquefy, warm slowly to 70° to 90°F. making sure the drum is vented. Do not use live steam.

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Shipping:

Sodium Borohydride-SWS has been classed as a corrosive liquid under DOT regulations. Laboratory quantities are shipped in one-gallon, sealed, polyethylene "cubitainers" in a cardboard outer container, which is in turn packaged in an DOT-approved wooden box. Development and production quantities are shipped in 55-gallon drums, which have at least 10% free space. Larger quantities can be shipped by tank truck or tank car.

Safety:

Due to the presence of water, $NaBH_4$-SWS is considered non-flammable. Certain precautions must be taken, however. Borohydride solutions will decompose rapidly, evolving large amounts of hydrogen gas if subjected to acid conditions, excessive temperature, or the presence of metal salts or finely divided metallic precipitates such as nickel, cobalt or copper.

Toxicity:

Due mainly to the presence of caustic, NaOH, Sodium Borohydride-SWS is considered toxic if ingested. Borohydride solutions are also toxic if ingested primarily due to possible gas embolism resulting from reaction with stomach acids. All precautions should be taken to avoid direct contact or ingestion.

First Aid:

In case of accidental contact, flood with copious amounts of water. Wash skin with soap and water. Flush eyes with water. Seek medical attention promptly.

References:

VENTRON CORPORATION TECHNICAL BULLETINS:

1. Sodium Borohydride/Handling/Uses/Properties.
2. Inorganic Reductions with Sodium Borohydride.
3. Hydride Chemicals for Process Stream Purification.

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Ventron Corporation/Chemicals Division
Congress Street, Beverly, Massachusetts 01915/Tel: (617) 922-1875

DEVELOPMENT PRODUCT | OMH-1

SODIUM ALUMINUM DIETHYL DIHYDRIDE

$NaAl(C_2H_5)_2H_2$

APPLICATIONS

OMH-1 (sodium aluminum diethyl dihydride) is an excellent reducing agent which is soluble in aromatic hydrocarbons. It is similar in action to $LiAlH_4$ and $NaAlH_4$ and reduces a wide variety of functional groups in high yield without participation by the $-C_2H_5$ groups present. Most reductions occur readily at 25° C although temperatures above 100° C can be used with non-reactive solvents. OMH-1 should be useful for pharmaceutical, flavor and fragrance, and fine chemical applications requiring an active hydrogen reducing agent.

AVAILABILITY

Development quantities from one pint to 355 gallon portable tanks are available as a 25% solution in toluene containing 3-4% THF.

SOLUBILITY

OMH-1 is soluble in aromatic hydrocarbons and ethers. It is insoluble in paraffin hydrocarbons and reacts with hydroxylic or active hydrogen solvents.

PHYSICAL DATA

Form	25 wt% solution in Toluene*
Appearance	slightly colored solution
Typical Analysis	
Al, wt%	5.9
Active Hydride, meq/g soln	4.4
Total Gas Evolution, mmols/g soln	8.8
Density, g/ml at 20° C	0.879
lb/gal at 20° C	7.35
Viscosity, cp at 20° C	2.08

* Containing 3-4% THF.

HANDLING PRECAUTIONS

OMH-1 should be kept from contact with air (O_2) and moisture to prevent loss of reducing capacity through oxidation or hydrolysis. Solutions of greater than 10% by weight concentration are classed as pyrophoric for shipping purposes. However, concentrations of 10-25% by weight can be handled easily and safely if ordinary precautions are observed. Handling under an inert atmosphere is recommended. Rags, towels, or other combustible absorbent materials should not be used on large spills since ignition of the solvent may result under certain conditions. Dry chemical extinguishers are recommended for use on fires. Solid OMH-1 or its solutions are corrosive to human tissue, and adequate protection against contact with the skin and eyes should be employed during handling and use. The toxicological properties of solutions of OMH-1 have not been fully investigated. In case of accidental spills, inhalation of vapors or fumes should be avoided.

The information presented herein is believed to be accurate and reliable, but is presented without guarantee or responsibility on the part of Ethyl Corporation. Further, nothing contained herein shall be taken as an inducement or recommendation to manufacture or use any of the herein described materials or processes in violation of existing or future patents.

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FORM CDH :
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